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INFLUENCE OF THE COMPACTION PRESSURE ON THE TIB$_2$ SAMPLE INTEGRITY

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Abstract: *The goal of this paper is to determine the optimum pressure of compacting nanocomposite TiB_2 powders using FEM. The samples will be further sintered using resistive and microwave heating. The range of the particle size distribution of TiB_2 powders was between 0.14-0.37 µm. Three different values for compaction pressure were applied namely 500, 700 and 1000 MPa. The conclusion was that the pressure of 1000 MPa leads to the damage of the active element of the die. The finite element analysis offers profound insights into the structural behavior and material strength during the titanium diboride pressing process, providing essential information for the optimization and improvement of manufacturing technologies.*
Key words: *compaction pressure, TiB_2 nanopowders, FEM.*

1. INTRODUCTION

Titanium Diboride (TiB_2) powders represent a class of advanced materials garnering significant attention in various industrial and scientific domains. Renowned for its exceptional hardness, thermal stability, and electrical conductivity, TiB_2 is a ceramic compound that belongs to the family of diborides. Its use in cutting tools, aerospace components, and protective coatings illustrates its significance in advanced manufacturing. Additionally, TiB_2 plays a role in the development of high-performance ceramics and electronic devices, highlighting its versatility in various technological domains.

Powder compaction is an important process in materials science and engineering, playing a significant role in the production of various components across diverse industries. Cold powder compaction, specifically, involves forming a coherent mass from a powder at ambient or slightly elevated temperatures without the application of external heat.

As evidenced in the existing literature [1-3], it is well established that the green compaction process exerts a significant influence on the subsequent sintering behavior of TiB_2 composites. During compaction, details like the pressure applied, the nature of the powder, and the resulting green density significantly influence how the final composite material will behave once it undergoes sintering. Understanding and optimizing the green compaction process are imperative for achieving desired sintering outcomes. The compacted green body serves as the precursor for the subsequent sintering phase, affecting not only the densification kinetics during sintering but also the final microstructure and mechanical properties of the TiB_2 composite.

Building upon the findings of previous researches on sintering utilizing microwave heating [4-9], the intricate relationship between the stability of the process and the shape, as well as the uniformity of the green compact surface, has been emphasized. These investigations underscored that even minor surface defects within the sample can induce localized overheating, posing a potential risk to the structural integrity of the final product.

Moreover, preliminary examinations focusing on the compaction process of TiB_2 powders at lower pressures have revealed the inherent difficulties in achieving samples that meet the necessary criteria for subsequent microwave sintering. Recognizing the critical need for a comprehensive understanding of the

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compaction dynamics, a finite element analysis was employed, utilizing the simulation module embedded in the SolidWorks software. This analytical approach aimed not only to unravel the complexities of the compaction process but also to provide valuable insights guiding the refinement of sample preparation methodologies for optimal results in the subsequent microwave-sintering phase.

This combined approach of empirical investigations and advanced numerical simulations represents a concerted effort towards overcoming the challenges associated with microwave sintering of TiB_2 powders, offering a robust foundation for further advancements in the optimization of this intricate manufacturing process.

2. MATERIALS AND METHODS

The raw material employed in this study was commercially TiB_2 powder procured from Sigma-Aldrich, exhibiting a particle size of approximately 1 micron and a purity exceeding 99%.

There were also used very fine powders of TiB_2, resulted after mechanical milling, with a particle size distribution between 0,14-0,37 µm. The entire process of mechanical milling of TiB_2 powders is presented in the papers [10, 11].

Commercial and milled powders are presented in figure 1. TiB_2 powders were subjected to compaction process through the utilization of a cylindrical mold featuring an inner diameter of 15 mm, applying several pressing forces equivalent to 100, 200, 300 and 400 MPa.

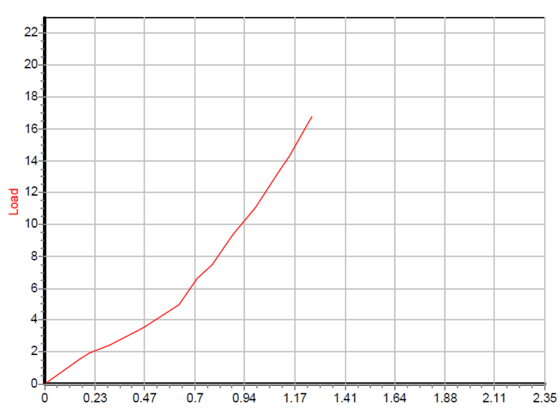
The compaction procedure was conducted using the LBG Universal Testing Machine, manufactured by LBG SRL, Italy, in the year 2010. The powder pressing process was unidirectional with a simple action, characterized by the fact that both the mold and the lower punch are fixed, the pressure being applied through the upper punch that penetrates the mold.

The equipment used for the compaction process is presented in figure 2, while the compaction curves corresponding to all the previously mentioned pressure values are shown in figure 3.

a) b)

Fig. 1 TiB_2 powders: a) commercial; b) milled

Fig. 2 Compaction equipment LBG Universal Testing Machine



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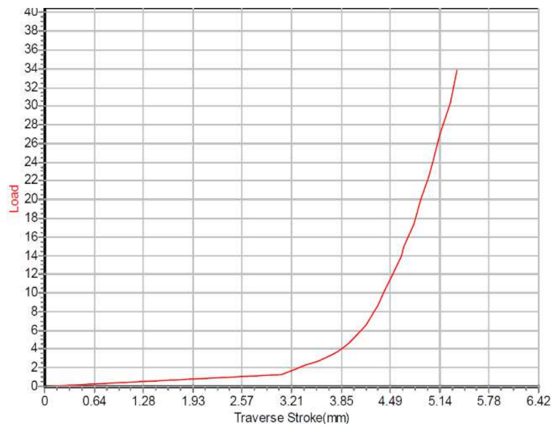

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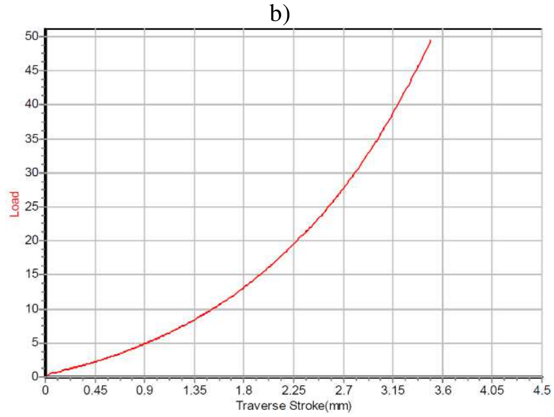

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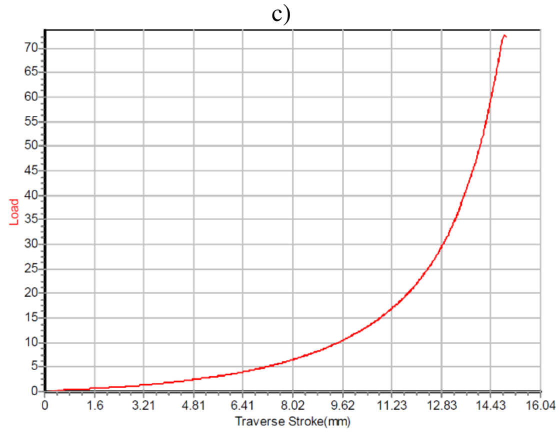


d)

Fig. 3 Compaction curves for TiB_2 powders: a. 100 MPa, b. 200 MPa, c. 300 MPa, d) 400 MPa

It is known that TiB_2 is a hard compound and its integrity as a green compact is very important for the microwave sintering process.

The preliminary researches on the compaction process of TiB_2 powders at low pressures (100-200 MPa) showed that it is hard to obtain samples suitable to be sintered using microwave heating. When removing the tablet from the mold, the samples lost their integrity, resulting in a series of defects as in figure 4.

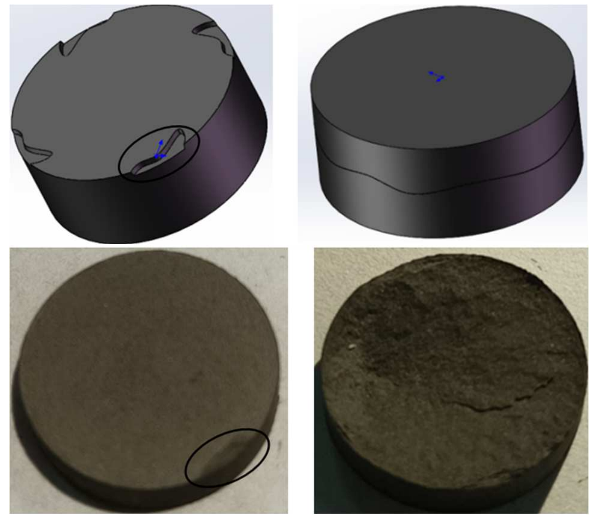


Fig. 4 Defects occurred during sample handling

When the pressure increased, the samples cracked either longitudinally or diagonally when there were handled. There were also situations when the powder stuck to the active part of the upper punch when it was extracted from the mold (figure 5).

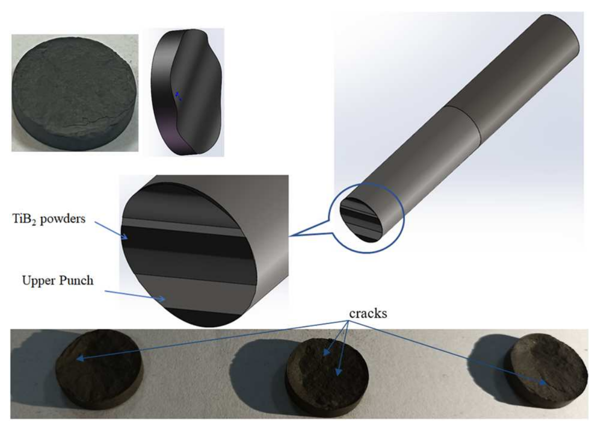


Fig. 5 Defects occurred when the sample was extracted from the mold

One of the explanations for the appearance of cracks in the TiB_2 samples can be the elastic relaxation phenomenon manifested when the tablet from the mold is removed, which causes an increase in the dimensions of the sample both in the direction of pressing and perpendicular to it, and there is a danger of it cracking upon ejection. In order to avoid these unwanted phenomena and to obtain a total integrity of the TiB_2 sample, a lubricant in the form of a powder $Zn(C_{18}H_{35}O_2)_2$ was used, which reduces the powder-mold friction, improves the compressibility of the powder and reduces by 3 to 5 times the pressure required to eject the sample. It is known that the cohesion of the sample is obtained by the direct contact between the powder particles. So, this lubricant was mixed with the TiB_2 powder in a proportion of 0.2 % and also was placed on the active parts of the punch and die. With the application of the previously mentioned, the compaction process was improved, but not completely. Although the powder no longer adhered to the punch, small cracks still formed. In order to solve this problem too, the compaction pressure was raised to 400 MPa.

For the mold used in the compaction process, it was a challenge to increase the pressure, but also to use mechanically milled TiB_2 nanometer powders. These very fine powders entered between the punch and the mold and led to their blocking and at the same time to the damage of the punches and the mold.

To continue the experimental activities, a new mold was designed with the dimensions shown in figure 6. The material for the active elements was C80U tool steel.

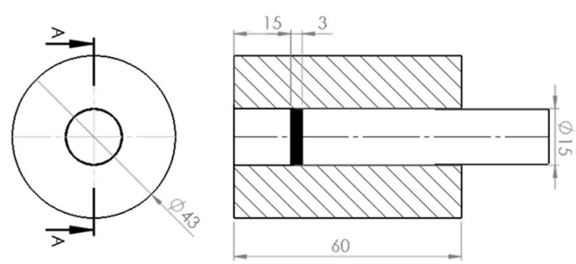


Fig. 6 2D Design of mold and punches

Specific thermal treatments were applied to both the mold and the punches.

In order to see the maximum stress level of the new mold, the finite element method (FEM) was used using the mechanical simulation module of the SolidWorks 2016 software.

3. RESULTS AND DISCUSSIONS

The application of the Finite Element Method in powder compaction facilitates a detailed analysis of the mechanical behavior and densification process, offering valuable insights into the compaction dynamics of powdered materials. Thus, the mold was fixed at the bottom (Fig. 7) and three pressures, namely 500, 700 and 1000 MPa, were applied to the sample. The material properties given include Young's Modulus of 286000 N/mm^2 and Poisson's Ratio of 0.28.

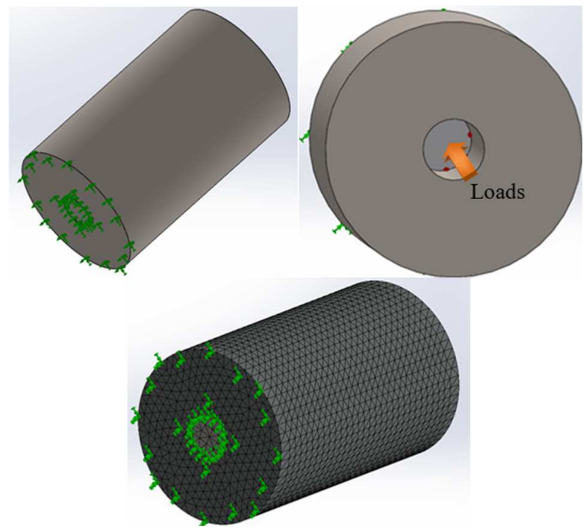


Fig. 7 Loads and meshed 3D model of mold

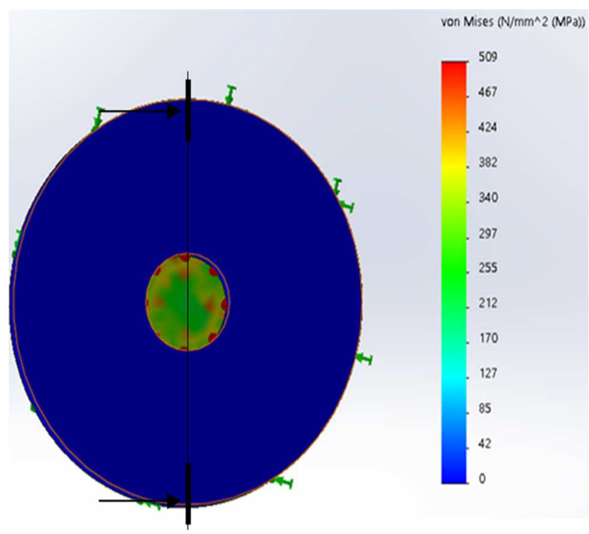


Fig. 8 von Mises stress state of the pressing process at 500 MPa

A first pressure load of 500 MPa was applied on the sample. The stress distribution is showed in figure 8.

The maximum intensity of stress was obtained at 509 MPa, which is less than the yield strength of C80U tool steel. In order to see the stress distribution in the mechanically stressed area, a section was made through the mold, as can be seen in figure 8.

In figure 9, the upper layer of the sample was investigated by its nodal scoring to see the distribution of von Mises stresses.

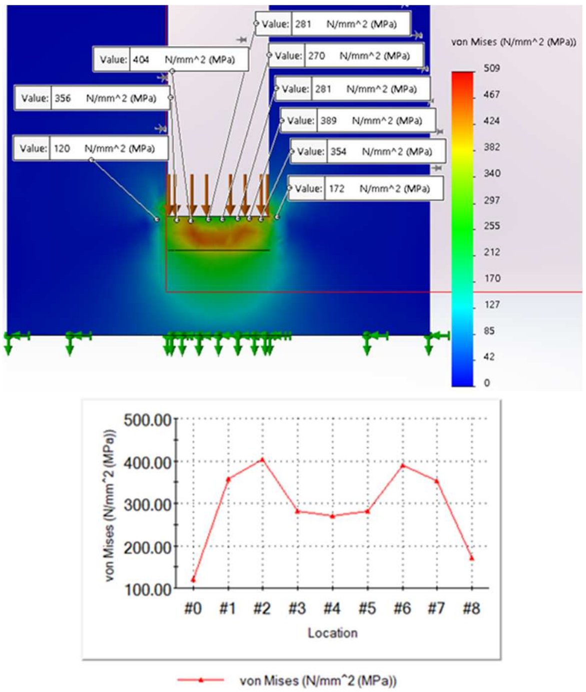
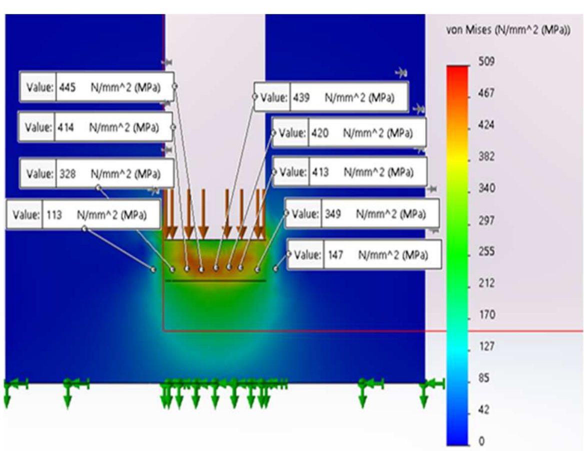


Fig. 9 von Mises stress in the upper layer of the sample for the compaction pressure of 500 MPa (section through the mold)

According to the graph, it can be seen that the stresses are higher at the edge of the piece than in the middle.

The von Mises stress pattern in the bottom layer of the sample is shown in figure 10.



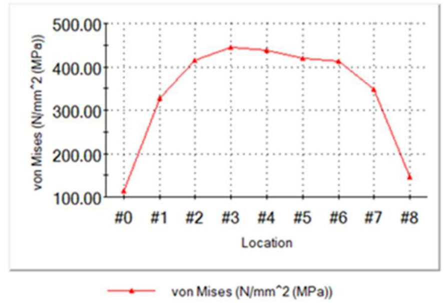


Fig. 10 von Mises stress in the bottom layer of the sample for the compaction pressure of 500 MPa (section through the mold)

The nodal-pointed longitudinal part at the bottom of the sample recorded higher stresses than the other two parts.

The stresses appearing in the lower punch were also investigated and it was found that they do not exceed the yield limit of the material from which it is made, according to figure 11.

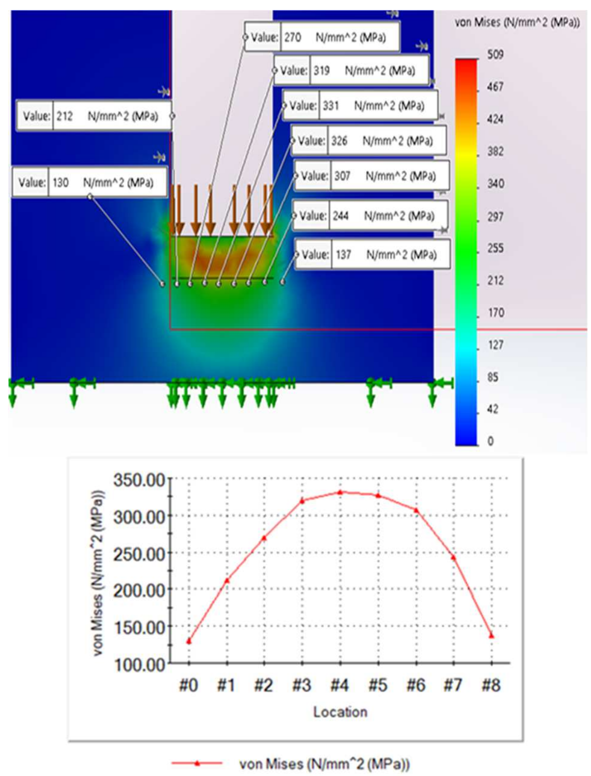


Fig. 11 von Mises stress in the active area of the lower punch for the compaction pressure of 500 MPa (section through the mold)

In figures 12 - 14 the upper and bottom layers of the sample were investigated to see the distribution of von Mises stresses for a compaction process of 700 MPa and also the stresses in the lower punch were investigated.

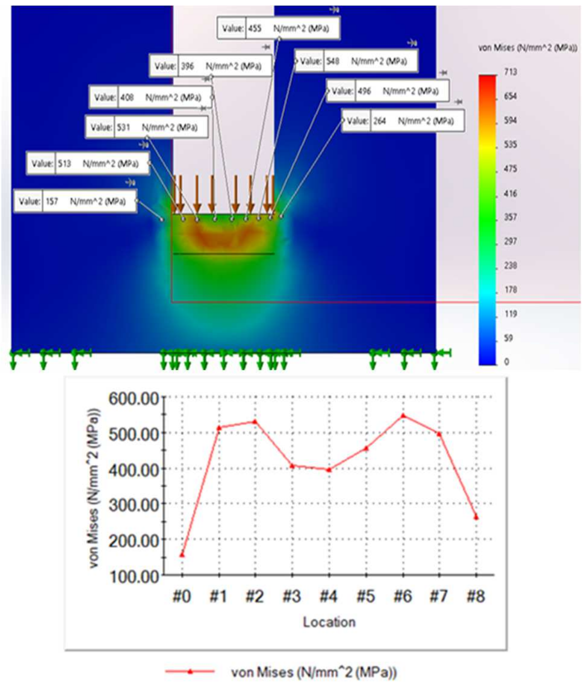


Fig. 12 von Mises stress in the upper layer of the sample for the compaction pressure of 700 MPa (section through the mold)

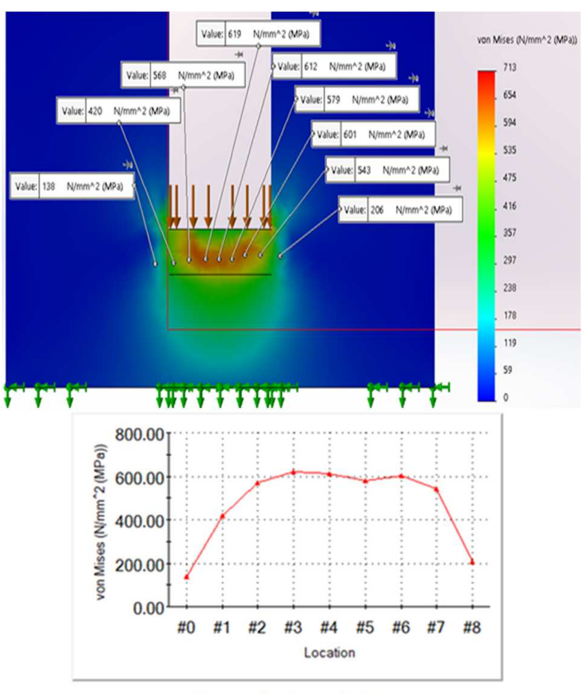


Fig. 13 von Mises stress in the bottom layer of the sample for the compaction pressure of 700 MPa

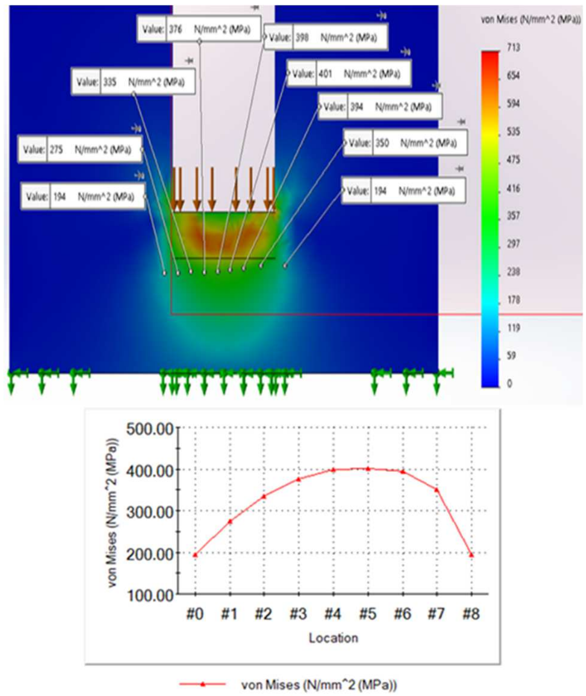
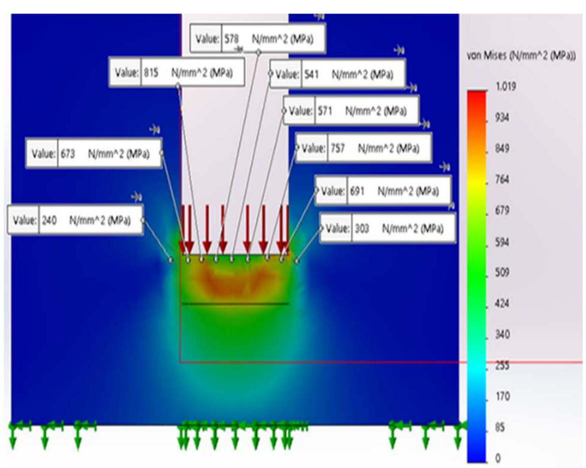


Fig. 14 von Mises stress in the active area of the lower punch for the compaction pressure of 700 MPa

Higher stresses were recorded in this case compared to the compaction pressure of 500 MPa. Even at this pressure of 700 MPa, there were no stresses above the yield limit of the material.

The simulation of the compaction process for a pressure of 1000 MPa was also carried out. The results are presented in figures 15 - 17.



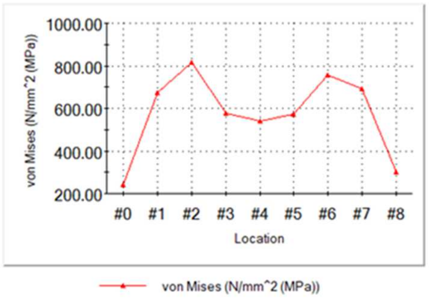


Fig. 15 von Mises stress in the upper layer of the sample for the compaction pressure of 1000 MPa (section through the mold)

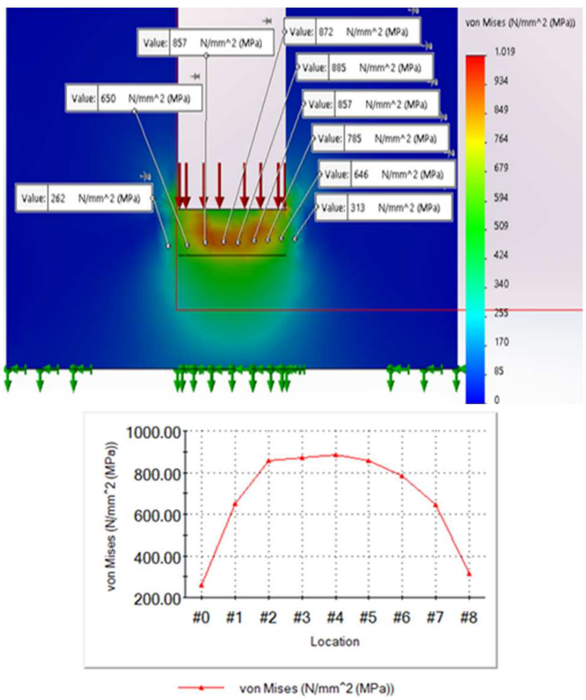
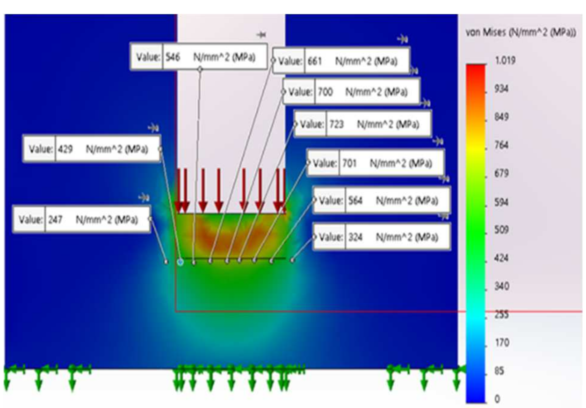


Fig. 16 von Mises stress in the bottom layer of the sample for the compaction pressure of 1000 MPa



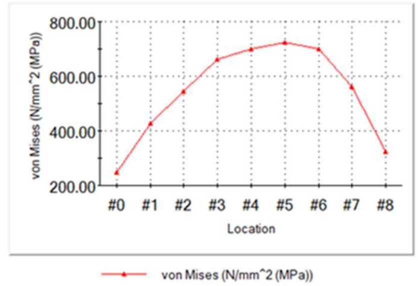


Fig. 17 von Mises stress in the active area of the lower punch for the compaction pressure of 1000 MPa

For the first two compaction pressures of 500 and 700 MPa, there were no stresses that exceeded the yield limit of the punches or the mold, on the other hand, at the compaction pressure of 1000 MPa, stresses above the yield limit were recorded at the lower punch.

In the compaction process, the behavior of the upper punch during mechanical stress is also important. That is why a compression testing of the upper punch was performed and there were applied two pressures, namely 500 and 700 MPa, according to the figures 18 and 19.

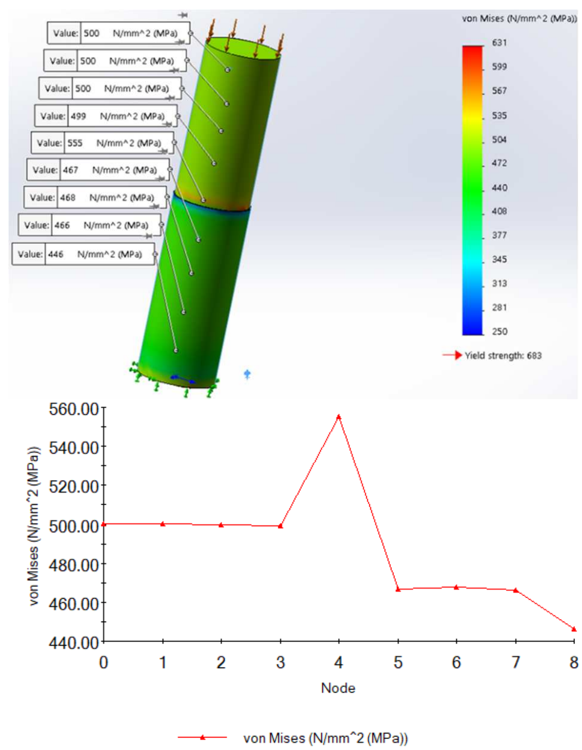


Fig. 18 Compression testing of the upper punch using a pressure of 500 MPa

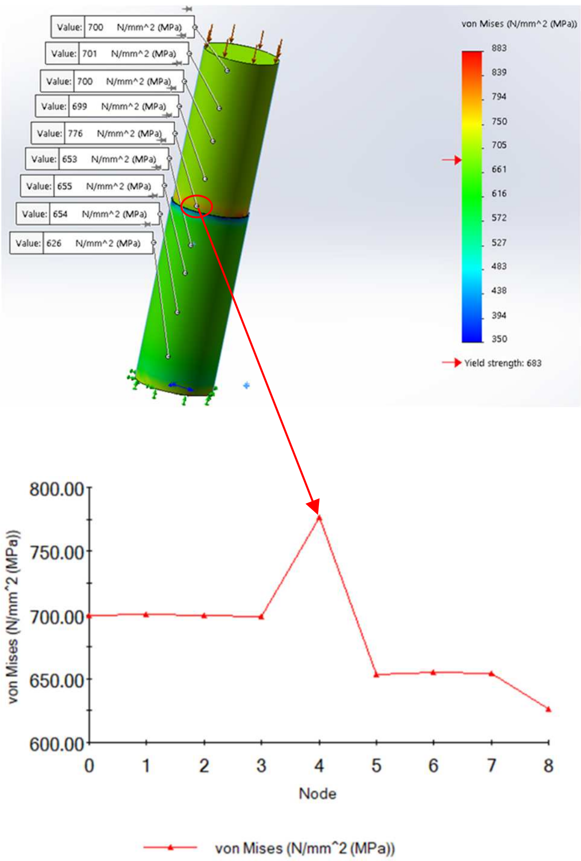
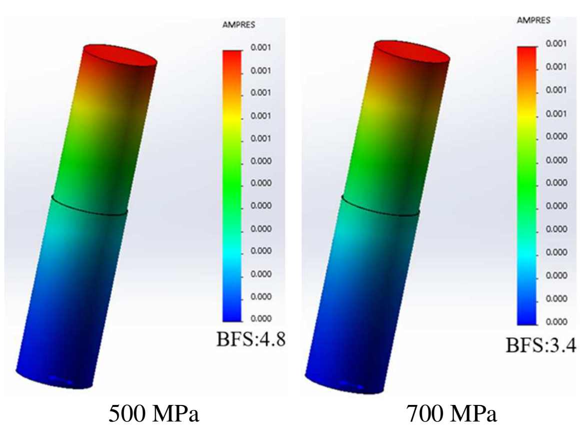


Fig. 19 Compression testing of the upper punch using a pressure of 700 MPa

It is evident that under a pressure of 700 MPa, remanent stresses emerge, localized in the central region of the punch, as presented in the graph from figure 19.

For a comprehensive analysis, a buckling test was conducted on the upper punch, subjecting it to pressures of 500, 700, 1000 and 2500 MPa, as illustrated in figure 20.



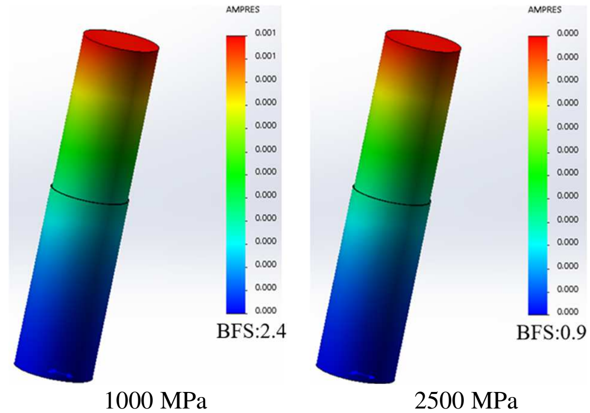


Fig. 20 Buckling testing of the upper punch

The buckling assessment indicated that the upper punch successfully passed the test until reaching 2500 MPa, beyond which it failed.

According to the finite element analysis, it was found that the maximum pressure applied to the TiB_2 powders must be up to 700 MPa.

So, the first pressure applied to the new mold was approximately 480 MPa. The lubricant was also applied to the active elements of the mold, and the result is the one in figure 21.

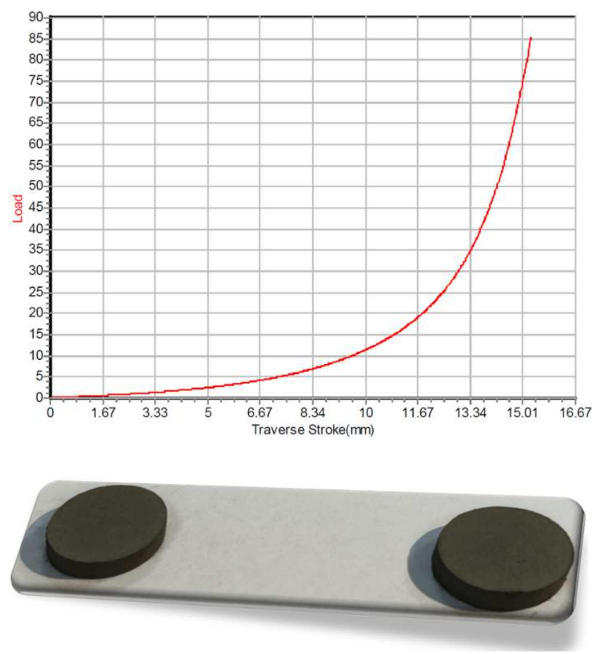


Fig. 21 Compaction curve for TiB_2 powders at 480 MPa and the obtained green sample

It can be clearly seen that the integrity of the TiB_2 samples is flawless.

4. CONCLUSION

It was found that the low compaction pressures of the TiB_2 powders do not help to consolidate the green sample.

Utilizing the Finite Element Method for powder compaction enables a comprehensive analysis of mechanical behavior and densification processes, providing valuable information about the dynamics of compacting powdered materials. The stresses exhibit a non-uniform distribution in the titanium diboride sample, with maximum values observed at the marginal regions and the lower layer of the specimen. The initial compaction pressures of 500 and 700 MPa did not generate stresses surpassing the yield limits of the lower punch or the mold. However, at a compaction pressure of 1000 MPa, stress values exceeding these limits were recorded, particularly at the lower punch. During the compression test of the upper punch, it failed to withstand a pressure of 700 MPa. The buckling test demonstrated that the upper punch withstood pressures up to 2500 MPa but ultimately failed past that threshold. These observations provide profound insights into structural behavior and material strength within the titanium diboride pressing process, offering important information for the optimization and enhancement of manufacturing technologies.

It can be mentioned that the optimal compaction pressure of fine TiB_2 powders is between 400 and 500 MPa. It is also indicated to apply the lubricant $Zn(C_{18}H_{35}O_2)_2$ on the active elements before compaction process.

5. ACKNOWLEDGEMENTS

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Influența presiunii de compactare asupra integrității probelor de TiB_2

Rezumat: Scopul acestei lucrări este de a determina presiunea optimă de compactare a pulberilor nanocompozite de TiB_2 utilizând metoda elementelor finite (FEM). Probele vor fi ulterior sinterizate folosind încălzire rezistivă și cu microunde. Distribuția dimensiunii particulelor pulberilor de TiB_2 s-a situat în intervalul 0,14-0,37 µm. Au fost aplicate trei valori diferite pentru presiunea de compactare, și anume 500, 700 și 1000 MPa. Concluzia a fost că presiunea de 1000 MPa duce la deteriorarea elementului activ al matriței. Analiza cu elemente finite oferă perspective profunde asupra comportamentului structural și a rezistenței materialului în timpul procesului de presare a diboridului de titan, furnizând informații importante pentru optimizarea și îmbunătățirea tehnologiilor de fabricație.

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