

Article

Structure, Phase Composition, and Properties of Ti_3AlC_2 —Nano-Cu Powder Composites

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Abstract: Composites based on the MAX-phases are promising materials for wide range application. Composites MAX-phase–copper can be used in electrical engineering as wear-resistant and durable sliding contact materials. Such composites can be used as coatings on sliding contacts to improve local strength and wear-resistance without a significant increase in production costs. In this work, Ti_3AlC_2 —nano-Cu composites with the ratio $\text{Ti}_3\text{AlC}_2\text{:Cu} = 1\text{:}1$ by weight or approximately 4:1 by volume were studied. The main task of the study is to obtain a dense structure, as well as to study the effect of the sintering temperature of the samples on their structure, phase composition, mechanical properties, and electrical conductivity. In addition, the sintered specimens were subjected to a hot isostatic pressing to possibly further increase the density. It was found that the best combination of strength, density, and electrical conductivity is achieved after sintering at 1050 °C. A further increase in the sintering temperature leads to an intensification of the MAX phase decomposition process, and at a lower sintering temperature, the copper matrix remains incompletely formed.

Keywords: MAX-phase; nanopowder; copper; hot isostatic pressing



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1. Introduction

Ternary carbides and nitrides with formula $\text{M}_{n+1}\text{AX}_n$ ($n = 1, 2$, and 3, M is an early transition metal, A is an A group element, and X is C and/or N) called MAX-phases are promising materials because of the unique combination of both metals and ceramics properties [1]. The Ti_3AlC_2 MAX-phase has a low density, high modulus of elasticity, high strength at high temperature, good workability, good oxidation resistance, excellent thermal, electrical conductivity, and so on. MAX-phases can also be transformed into MXenes using an etching process. The first explored application of MXenes was in energy storage, which remains a large proportion of MXene activities. MXenes is also a promising material for electromagnetic applications in [2–5]. Thus, MAX-phases can improve mechanical properties by acting on the mechanism of a hardening secondary phase [6–9] and also affect the electrical properties of the material when using MXenes.

It is difficult to obtain bulk products from the MAX-phase powders by powder metallurgy methods, in particular, due to the decomposition of the MAX-phase at elevated temperatures [10–12]. Due to the fact that the MAX-phase has a layered structure, and its high-temperature molding is impossible due to decomposition processes, products from the MAX-phase are predominantly porous. One of the promising methods for obtaining dense products based on the MAX-phase is the production of metal matrix composites (MMCs). In such composites, the metal acts as a binder for the MAX-phase and also fills the pores.

A promising MMC with a MAX-phase is a composite with a copper matrix [13–15]. Such composites have increased electroerosion resistance, ablation resistance, and high conductivity, which makes these materials promising for use in electrical engineering.

In particular, MAX-phase–Cu composites are suitable as materials for high-speed train pantographs, since MAX-phase addition to copper can improve its strength, hardness, and wear resistance without affecting its self-lubrication and electrical conductivity [16]. The composite does not have to be used in bulk form; to reduce the cost, protective coatings can be made on working parts in the contact zone.

In this work, Ti_3AlC_2 –nano-Cu composites with the ratio $\text{Ti}_3\text{AlC}_2\text{:Cu} = 1\text{:}1$ by weight or approximately 4:1 by volume were studied. The main task of the study is to obtain a dense structure, as well as to study the effect of the sintering temperature of the samples on their structure, phase composition, mechanical properties, and electrical conductivity. In addition, the sintered specimens were subjected to a HIP treatment to possibly further increase the density. Investigated materials can be an effective surface modification of sliding contacts. Copper was chosen as the matrix because the coating is planned to be applied to copper. MAX-phases are chosen as hardening particles, while not significantly reducing the overall electrical conductivity and having the properties of a solid lubricant. To date, such composites are used for pantographs in [17]. Our task in the future is to develop materials of composition Ti_3AlC_2 –Cu suitable for extrusion 3D printing (material extrusion additive manufacturing, MEAM).

2. Materials and Methods

2.1. Samples Preparation

The Ti_3AlC_2 MAX-phase powder (Jiangsu XFNANO Materials Tech Co., Ltd., Nanjing, China, 98% purity, $d_{50} = 5 \mu\text{m}$) was used in this work. Nanosized copper powder was obtained by the electric explosion of wire (EEW). The use of nanosized copper in our work is due to the technological features of the subsequent process of forming parts. The methodology for obtaining powders, as well as their characterization, is described in the article [18,19].

Mixing of the powders was carried out by the dry method in turbula-type mixer (Turbula S2.0, Vibrotechnik JSC, Saint-Petersburg, Russia). Powder mixtures of Ti_3AlC_2 and Cu were prepared with a ratio of 1:1 by weight within an hour. The resulting mixtures were pressed in a cylindrical die $\varnothing 20 \text{ mm}$ at a load of 5 tons.

The samples were sintered in a vacuum furnace (Nabertherm GmbH, Lilienthal, Germany) at temperatures from 800 to 1100 °C. The heating rate was 5 K/min, and the isothermal holding at the sintering temperature was 2 h. Experiments were also carried out on hot isostatic pressing (HIP) of the obtained samples after sintering. HIP was carried out at a temperature of 1000 °C for 22 h at a gas pressure of 1600 bar. Samples after vacuum sintering at 900 and 1000 °C were used for HIP.

2.2. Samples Investigation

The microhardness of the samples was measured on a microhardness tester (Duramin-500, Struers, Copenhagen, Denmark) by the Vickers method (HV) at a load of 200 gf. The samples were studied by optical metallography (Axiovert 200-MAT, Carl Zeiss GmbH, Jena, Germany) and scanning electron microscopy (Apreo S, Thermo Fisher Scientific, Madison, WI, USA). For the study, cross-sections were prepared on a disc grinding–polishing machine (Saphir 520, ATM Qness GmbH, Mammelzen, Germany) using SiC paper with 400–1600 mesh size and diamond discs with 2000–4000 mesh size.

The phase composition of the samples was studied using an XRD-7000S X-ray diffractometer (Shimadzu, Kyoto, Japan) in the Bragg–Brentano configuration using $\text{CuK}\alpha$ radiation with a long wavelength $\lambda = 0.154 \text{ nm}$ at X-ray tube parameters of 40 kV and 30 mA. For each of the samples, a spectrum was collected with the following parameters: scanning step— 0.0143° ; sample scanning speed— $10^\circ/\text{min}$; and range of angles in 2θ — 30 – 90° .

The three-point bending test was performed on a Gotech AL-7000M setup with one impact and two supporting rollers. For bending testing, $4 \text{ mm} \times 4 \text{ mm} \times 18 \text{ mm}$ samples were cut from the specimens by electrical discharge machining (EDM). The Gotech data acquisition software was used to trigger the minimum load (0.1 N) on the sample and to

start a data collection. The acquisition frequency was 124 Hz. The loading rate was set up as 0.5 mm/min according to the ASTM standard (E290). The span distance was equal to 16 mm.

The electrical conductivity of the samples and their comparison with the international annealed copper sample (IACS) was carried out using a 34410A multimeter (Keysight Technologies, Santa Rosa, CA, USA), verified in January 2022. Segments 6 mm in diameter and 10 mm long were cut by the EDM method from cylindrical pressed samples. To verify the measuring method, measurements were taken on a copper wire 6 mm in diameter and 10 mm long, showing the 99% IACS conductivity.

3. Results and Discussion

3.1. Microstructure and Phase Composition

A series of sintering was carried out at different temperatures (Figure 1). It was found that, with an increase in the sintering temperature, the structure of the samples changes. There are almost no differences in the structure of specimens sintered at 800 and 900 °C since these temperatures are low for sintering in the $\text{Ti}_3\text{AlC}_2/\text{Cu}$ system, and the material remains almost unchanged. The basis for selecting the sintering temperature is copper; in order to achieve a high density, it is necessary to focus on its sintering/melting temperature. During sintering at 800 °C, large voids are present in the samples, and unsintered copper particles are also found. During sintering at 900 °C, almost no such artifacts are observed.

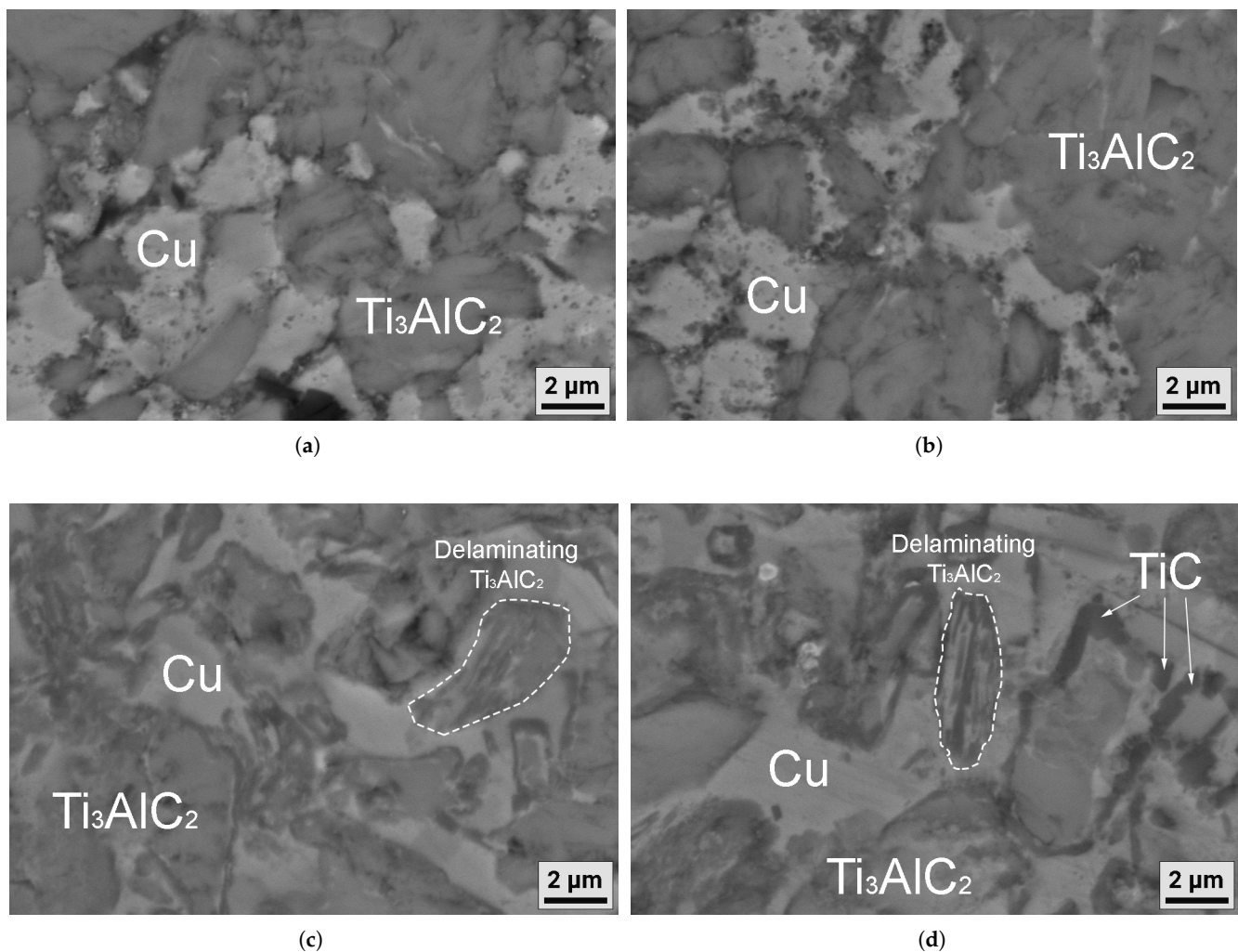


Figure 1. SEM pictures of Ti_3AlC_2 : Cu (1:1) cross-sections after sintering at 800 (a); 900 (b); 1000 (c); and 1050 (d) °C in vacuum.

Increasing the sintering temperature to 1000 °C leads to changes in the structure of the samples and the MAX-phase begins to decompose, which leads to the formation of delaminated particles of the MAX-phase. A shell of carbide particles is formed on the surface of large Ti_3AlC_2 particles and copper penetrates into some especially strongly exfoliated particles.

A structure corresponding to a metal–matrix composite is formed; particles or agglomerates of the MAX-phase are located in a copper matrix (Figure 2). The melting point of copper is 1085 °C [20], so sintering intensifies when approaching this temperature. In addition, the presence of copper nanoparticles may also have an effect, since the melting point of nanoparticles is below micron materials [21,22]. This is indicated by the presence of a liquid phase during sintering in some samples sintered at 1000 °C. However, no weight loss was observed for the samples. Sintering above the melting temperature causes the copper to flow out of the material, either partially or completely. In this regard, it is optimal to carry out sintering at a temperature not higher than 1050 °C.

Carrying out experiments with micron powder (Figure 3a) shows that the structure of samples during sintering at 1000 °C differs from the structure of samples with nanopowder (Figure 3b). When using samples with micron powder, the intensity of decomposition of the MAX phase is higher. In the studied samples with micron powder, there are few initial particles of the MAX phase left. In materials with a nanopowder, the amount of the initial MAX phase is much higher: the decomposition process is just beginning and is mainly observed on the surface of MAX phase particles.

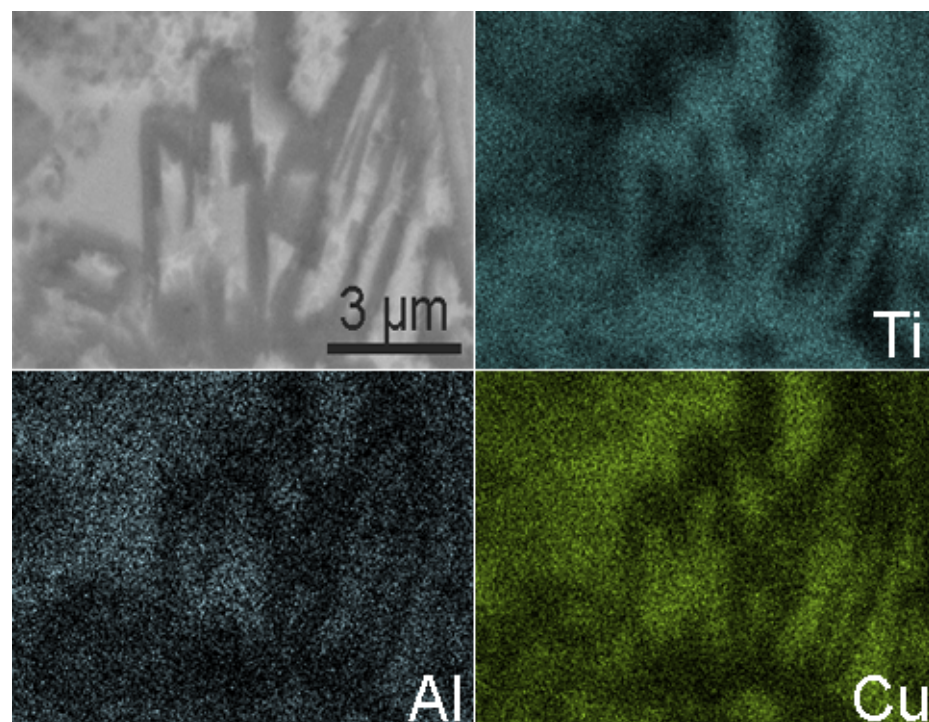


Figure 2. EDX maps of Ti, Al, and Cu for sample after sintering at 1050 °C.

The presence of MAX-phase decomposition is inevitable during high-temperature sintering. XRD phase analysis (Figure 4) shows that, in all samples, the MAX phase is present in the sample in an amount sufficient for detection by XRD. After 1000 °C, very intense TiC peaks are present in the XRD patterns. The copper oxide phase is also detected in all samples along with pure copper. Copper nanoparticles are very active and form an oxide very actively [23–25].

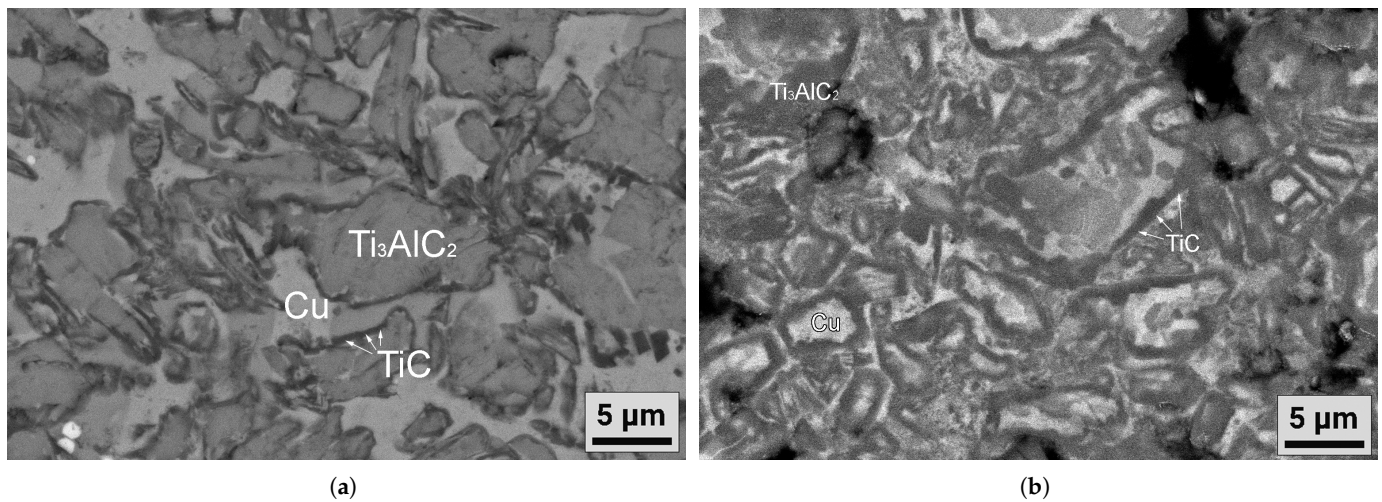


Figure 3. SEM pictures of Ti_3AlC_2 : Cu (1:1) cross-sections of the samples with nano (a) and micro (b) copper powder after sintering at 1000 °C in vacuum.

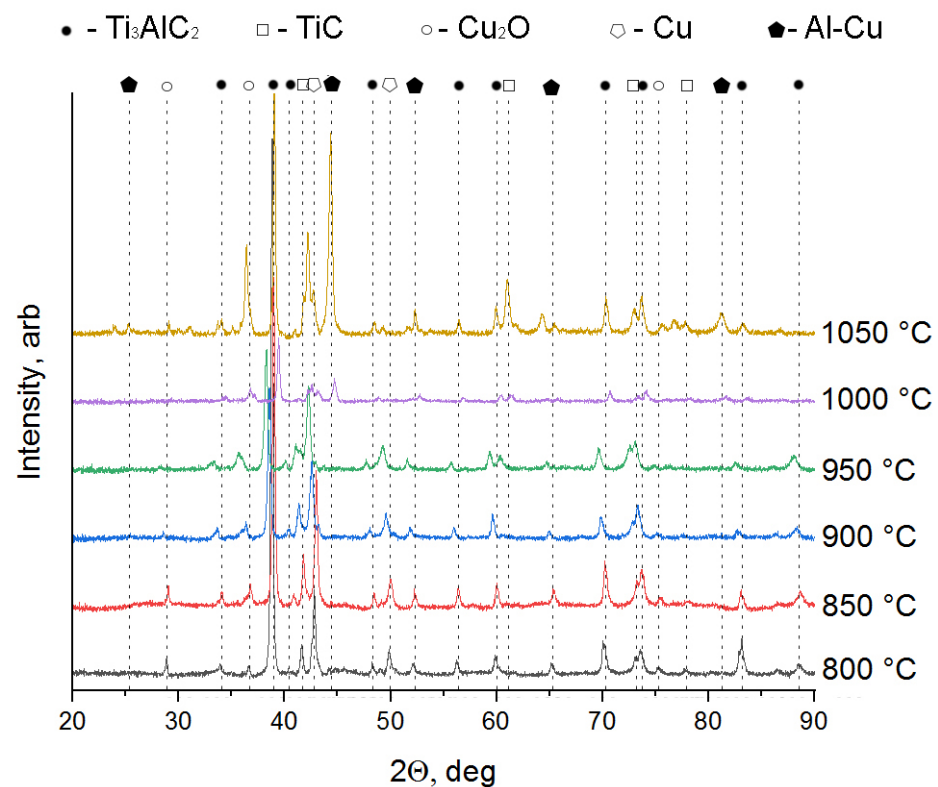


Figure 4. XRD patterns of Ti_3AlC_2 : Cu (1:1) samples after sintering at different temperatures.

The oxide formation does not stop after forming an oxide film on a surface as for Al [26,27]. Thus, the use of copper nanopowder leads to the presence of copper oxide in the material. Copper oxide is partially reduced during sintering but is present in the samples after sintering at all temperatures studied. Reflections from the intermetallic compound of copper and aluminum are also detected on the XRD patterns. The most intense reflections appear after sintering at 1000 °C and above.

After 1000 °C, the active decomposition of the MAX phase occurs. The aluminum contained in the MAX phase leaves the particles and enters the copper, since the particles of the MAX phase are located in the copper matrix. Thus, after leaving the MAX phase, aluminum forms intermetallic compounds in the presence of a copper matrix. In the

absence of a matrix, it decomposes into TiC and TiAl [28,29]; during high-temperature synthesis, aluminum may flow out or evaporate.

3.2. Porosity

Since the purpose of the work is to obtain fully-dense samples, the density of the samples was measured (Figure 5a). With an increase in the sintering temperature, the density of the samples increases, which is associated mainly with the sintering of copper. Sintering is accompanied by sample shrinkage. The higher the sintering temperature, the higher the shrinkage. Linear shrinkage increases from less than 1% for sintering at 800 °C to 9% for sintering at 1050 °C.

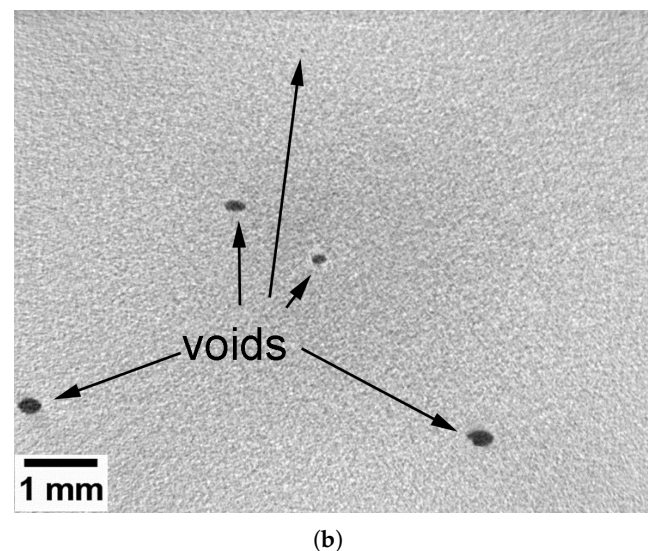
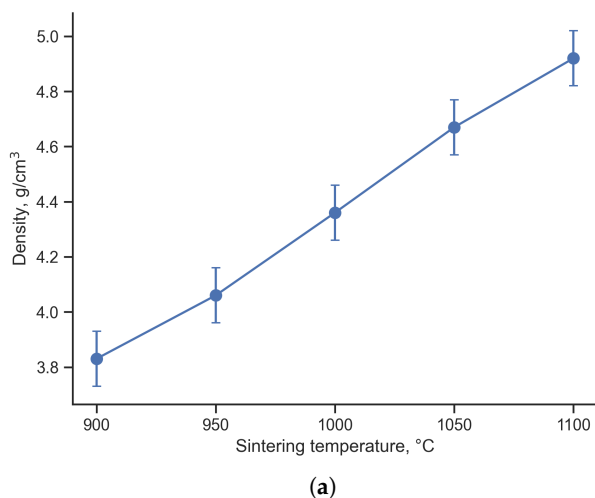


Figure 5. Density of Ti_3AlC_2 : Cu (1:1) samples after sintering at different temperatures (a) and typical X-ray tomography slice image (b).

The calculated density makes it possible to calculate the porosity of the samples; however, for such a calculation, it is necessary to know the theoretical density of the material. Calculation of the theoretical density of samples in this system is difficult, since phase changes occur (decomposition of the MAX phase, formation of the Al-Cu intermetallic compound, and others). In addition, during sintering at 1100 °C, molten copper may flow out and the ratio between the components may change. When using the theoretical density of a system with two components (Ti_3AlC_2 and Cu) in a 1:1 mass ratio, the minimum calculated porosity is 10%.

Non-destructive testing of the samples was carried out by X-ray tomography. The detected voids correspond to a porosity of no more than 3% (Figure 5b). There are several reasons explaining the high porosity obtained by the calculations (10% and above). The first reason is the presence of other phases that affect the density. It is known that TiC phases, Ti-Al intermetallic compounds, and Al-Cu solid solution are formed in the material after sintering. The difficulty lies not only in determining the exact amount of a particular phase in the material, but also in determining its density. Thus, for the Al-Cu solid solution, depending on its composition, the density differs significantly [30,31].

In addition, it is necessary to take into account in the calculations how many initial components it took to form new products. It is nearly impossible to determine this, since the reactions that occurred during the formation of the components are not clear. In addition, the SEM images show that the discontinuities and cracks in the structure of powder agglomerates are a few microns or less. An X-ray tomography is not capable of detecting defects smaller than 5–10 μm for the material under study under the selected imaging parameters. Thus, many voids left undetected. Based on this, it can be assumed that the sample is rather porous, and, according to the SEM, the main voids are formed

inside the MAX phase agglomerates. The density of specimens with micron copper during sintering below the melting temperature is even lower (3.9 g/cm^3 after sintering at 1000°C), which indicates its significantly higher porosity. In this case, the contribution to a porosity comes from copper particles, which, due to their size, do not penetrate deeply into the MAX phase agglomerates.

Samples after sintering at 900 and 1000°C were subjected to HIP. The samples were measured for Vickers hardness and density before and after HIP. It was found that the hardness of the samples after HIP treatment remained unchanged, and the density increased from 3.83 (900°C) and 4.36 (1000°C) g/cm^3 to 4.45 (900°C) and 4.51 (1000°C) g/cm^3 , respectively. The density of samples HIP-ed after sintering at 1000°C does not exceed the density of samples sintered 1050°C without HIP. The density of specimens sintered at 900°C after HIP increases more than that of specimens after 1000°C . This is explained by the fact that HIP leads to the sintering of samples and “healing” of voids due to pressure. Since HIP was carried out at 1000°C , samples sintered at 900°C were additionally sintered at HIP. Samples after 1000°C were mainly compacted due to pressure. Since the samples have a relatively high porosity, it is not possible to create a significant pressure on the voids inside the samples. In addition, the sample contains voids larger than $10 \mu\text{m}$, which are difficult to “heal” by HIP. Thus, for such samples, the HIP method is not optimal, since an increase in the sintering temperature without the use of HIP leads to a greater compaction of the samples. The density of the samples depends more on the kinetics of copper sintering, which is determined mainly by temperature rather than pressure.

3.3. Electrical and Mechanical Properties

The electrical conductivity of the studied materials increases with increasing sintering temperature and reaches 75% IACS at 1050°C (Figure 6). The trend is related to copper sintering and specimen densification. Sintering copper and forming a solid copper matrix leads to the increase of conductivity by forming more solid contacts, reducing porosity, and reducing the number of copper–copper interfaces. In this case, the MAX phase reduces the overall conductivity of the composite.

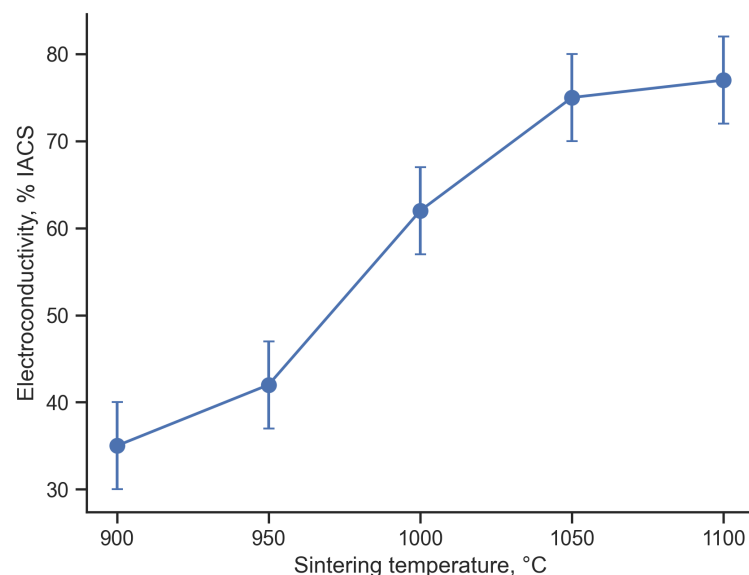


Figure 6. Electroconductivity in terms of % IACS of $\text{Ti}_3\text{AlC}_2 : \text{Cu}$ (1:1) samples.

The increase in electrical conductivity slows down after reaching the sintering temperature of 1000°C , despite the denser sintering of copper and an increase in the overall density at 1050°C . This is explained by the intensification of the decomposition of the MAX phase at this temperature. The most negative consequence of this is the formation

of titanium carbide (TiC), which has a lower conductivity than the MAX phase. However, TiC-Cu composites have a conductivity comparable [32,33] to that obtained in this study.

The hardness of the samples increases with an increase in the sintering temperature. When comparing data obtained from a material with a ratio (by weight) Ti_3AlC_2 : Cu = 1:1 and 2:1 (Figure 7a), it was found that the hardness after sintering at 800 °C is nearly the same for both compositions. This indicates a low sintering strength at this temperature and hardness at this temperature depends on the presence of many weak interparticle contacts. With an increase in the sintering temperature, the hardness of both materials increases, and the hardness of the material with a ratio of 2:1 exceeds the hardness of the other sample for all temperatures. After sintering at 1050 °C, the hardness of the sample with ratio of 2:1 is two times higher than for a sample with a 1:1 ratio.

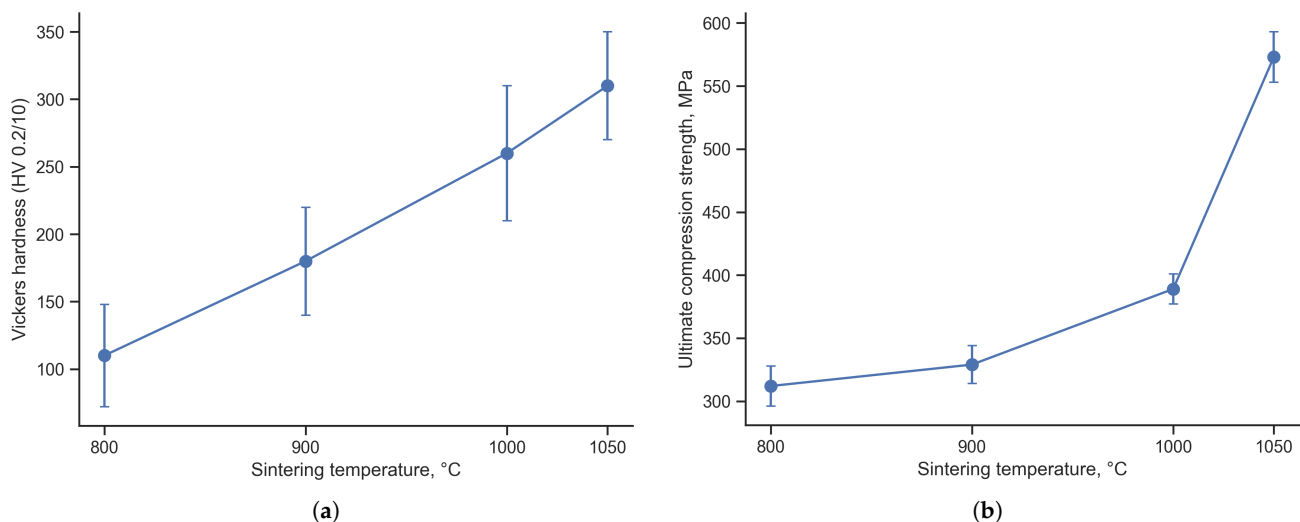


Figure 7. Vickers hardness (a) and ultimate compression strength (b) of Ti_3AlC_2 :Cu samples after sintering at different temperatures.

With an increase in the sintering temperature, a stronger and more continuous copper matrix is formed, and the effect of strengthening particles increases. Ti_3AlC_2 particles act as reinforcing particles, which is the reason for the higher hardness of samples with a ratio of 2:1. At high temperatures, the resulting titanium carbide particles also contribute to the strength, which is associated with a continuing increase in hardness [34–37]. Hardness, strength, and electrical conductivity are primarily determined by the copper matrix. Since we obtain products from powder materials, the strength of the matrix depends on how well the copper powder was sintered. However, inclusions of the MAX phase are important not only for strength and electrical conductivity, but for the effect of solid lubrication during the operation of sliding contacts [17]. Indeed, the hardness and strength of composites with copper will be higher if, for example, TiC or SiC is added. However, when making a contact from such a material, the counterbody will wear out extremely. In addition, the incorporation of TiC or SiC particles lowers the electrical conductivity below 50% IACS [38]. Thus, Ti_3AlC_2 should not be considered as a harmful impurity in the studied composites. The use of MX-enes could potentially lead to better results, but this is not included in the current study.

The compressive strength remains almost unchanged at a sintering temperature of 800–1000 °C. There is a slight increase in strength associated with the formation of a copper matrix. After sintering at 1050 °C, there is a noticeable growth in strength compared to the previous growth. At this temperature, the formation of a strong copper matrix nearly ends. The decomposition of the MAX phase leads to the formation of Al-Cu solid solutions in the contact regions of copper and the MAX phase. Titanium carbide is also formed as a result of a MAX-phase decomposition. These phenomena strengthen the composite—titanium carbide particles are hardening dispersed particles, and the formation of Al-Cu

solid solutions leads to a stronger interaction between copper particles and regions of MAX-phase particles.

The destruction of samples in all cases proceeds by a mixed mechanism. When approaching the maximum compressive strength, the samples begin to crack. Samples after sintering at a temperature of 800 °C are destroyed into small pieces less than 1 mm in size. The surface of the fragments is typical for ductile failure; there is an unsintered powder on the surface. With an increase in the sintering temperature, the fragments increase in size, and after sintering at a temperature of 1050 °C, the sample breaks into several very large fragments. The surface of the fragments also corresponds to ductile failure, but no unsintered powder is observed on the surface.

4. Conclusions

In this work, Ti_3AlC_2 —nano-Cu composites with the ratio $\text{Ti}_3\text{AlC}_2\text{:Cu} = 1\text{:}1$ by weight or approximately 4:1 by volume were studied. The composites with Cu micropowder and composites with the ratio $\text{Ti}_3\text{AlC}_2\text{:Cu} = 2\text{:}1$ by weight were investigated for a comparison. In order to obtain the optimal ratio of strength, density, and at the same time keep the phase composition close to the initial one, it is necessary to use a sintering temperature of 1000–1050 °C. It was found that strength, hardness, and electrical conductivity primarily depend on the quality of copper matrix sintering. Sintering at a lower temperature does not lead to the formation of a sufficient level of properties. The use of HIP also does not lead to a significant compaction of the material due to the large internal porosity. The main concentrator of porosity is the voids in the MAX-phase agglomerates. For further compaction of the material, it is necessary to conduct studies on the deagglomeration of the MAX-phase powder before manufacturing the product. The optimal sintering temperature for the composites studied is a 1050 °C. After sintering at 1050 °C, composites are formed with a hardness of 310 HV, a density of 4.67 g/cm³, an electrical conductivity of 75% IACS, and a compressive strength of 573 MPa. Titanium carbide (TiC) and solid solutions of the Al-Cu system are also formed in the composite in addition to the initial components.

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Conflicts of Interest: The authors declare no conflict of interest.

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