

High-purity Ti_2AlC Powder: Preparation and Application in Ag-based Electrical Contact Materials

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Abstract: Ag-based electrical contact is the "heart" of low-voltage switch, and the Cd toxicity has long been a haunting problem. It is the research focus of the low-voltage switch to find new environment-friendly electrical contact materials. Starting from the design of reinforcement for Ag-based electrical contact in this work, the high-purity Ti_2AlC powder (99.2%) was synthesized by a simple and fast pressureless technique. Ag/ Ti_2AlC composite electrical contact material was also prepared with homogeneous structure, good bonding between Ag and Ti_2AlC particles, high relative density (95.7%), moderate hardness (96HV), satisfactory electrical conductivity (low resistivity of 79.5 $n\Omega\cdot m$), and favorable arc erosion resistance (mass loss of 4.4% after 5610 arc discharging cycles). These excellent structure and properties of Ag/ Ti_2AlC composite are mainly attributed to the good thermal and electrical conductivity of Ti_2AlC and the good wettability between Ag and Ti_2AlC . This composite is expected to replace the conventional electrical contact materials in the future.

Key words: Ti_2AlC ; electrical contact; metal-ceramic composite; conductivity; wettability; arc erosion resistance

As a core component, Ag-based electrical contact is widely used in the low-voltage switching devices, such as contactors, circuit breakers, and relays^[1]. Contact material is exposed to the extremely high temperature and oxygen-enriched environment during the making and breaking of circuit, thus its performance directly determines the life of electrical appliances. In the process of contacting, contact is subjected to various working conditions with different current, voltage, temperature, humidity, *etc.* Consequently, properties such as high electrical and thermal conductivity, low contact resistance, moderate hardness, good machinability, high resistance to oxidation and arc erosion are the prerequisites for electrical contact materials^[2]. Ag/CdO has been the preferred material for the electrical contacts since the early 20th century, but the toxicity of Cd brings a series of serious problems^[3-4]. Current Cd-free contact materials (Ag/SnO₂^[5], Ag/ZnO₂^[6], Ag/CuO^[7], Ag/C, Ag/W^[8], Ag/Ni^[9], *etc.*) cannot replace Ag/CdO completely due to their various drawbacks in the aspects of structure and property. Thus, finding the adequate environment-

friendly reinforcement to replace Ag/CdO is urgent.

Recently, MAX phase materials (chemical formula of $M_{n+1}AX_n$, where M is early transition metal, A is mainly group A element, X is C or N), with combined properties of both metal and ceramic, received great attention from academia and industry^[10-11]. Ti_2AlC is one member of this MAX phase family with typically lamellar structure, light weight (4.11 g/cm^3), high electrical conductivity (resistivity of 0.36 $\mu\Omega\cdot m$), thermal conductivity (46 $W\cdot m^{-1}\cdot K^{-1}$), and excellent machinability like metals, good resistance to high-temperature and oxidation like ceramics^[11]. Ti_2AlC is considered as a potential reinforcement material, whose properties meet the requirement of Ag-based electrical contact materials. Over the past few years, Sun and Liu, *et al* successfully prepared Ag-based composites with the reinforcements of Ti_3SiC_2 ^[12], Ti_3AlC_2 ^[13-16], and Ti_2SnC ^[17-18], and systematically studied their phase, microstructure, and properties. Besides the basic properties of MAX phase family, Ti_2AlC has the outstanding resistance to oxidation. Hence, Ag/ Ti_2AlC (Ag/TAC) composite is expected to be the potential sub-

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stitute to Ag/CdO.

There are two key points need to be paid attention in the studying of Ag/TAC composites. First, the impure phases affect the density, structure, interface, and properties of the composites. Thus, preparing high-purity Ti_2AlC powder is particularly important before studying the Ag/TAC composite electrical contact materials. The synthesis of Ti_2AlC bulk and powder by using hot pressing (HP)^[19], hot isostatic pressing (HIP)^[20], spark plasma discharging (SPS)^[21], and self-propagated sintering (SHS)^[22] has been reported. These techniques, however, cost highly with complex process, and produce Ti_2AlC with many impure phases such as TiC, Ti-Al compounds, and Ti_3AlC_2 . In contrast, the pressureless sintering technique (PLS) is simple, efficient, and low-cost^[23], which is more meaningful for the practical application. In addition, the preparation, microstructure, interface bonding, and electrical contacting property of composite can be largely affected by the wettability between reinforcement and Ag matrix. Therefore, the wetting behavior of Ag and Ti_2AlC is a key issue for the research and application of Ag/TAC composites.

The purpose of this work is to synthesize the high-purity Ti_2AlC powder by using pressureless technique, and study the effect of process parameters on the purity and microstructure of Ti_2AlC . Then the wettability, microstructure, mechanical properties, electrical properties, and arc erosion resistance of Ag/TAC composites were studied, and its application potential were also discussed.

1 Experimental

1.1 Synthesis of Ti_2AlC powder

Powders of TiC (~5 μm , 99.0%), Ti (~50 μm , 99.99%), and Al (~50 μm , 99.7%) were weighed according to stoichiometric ratio and then were totally homogenized in the three-dimensional mixer (Turbula T2F, WAB, Switzerland) for 24 h. Mixture powders were directly heated to the setting temperature for 1 h at a heating rate of 10 $^{\circ}C/min$ under the protection of flowing Ar gas (99.999% purity).

1.2 Wetting test of Ag/ Ti_2AlC

Ti_2AlC bulk (a relative density of 99.6%) was prepared by using SPS (heated to 1300 $^{\circ}C$ at a heating rate of 50 $^{\circ}C/min$ and held for 20 min with a applied pressure of 50 MPa). The prepared composite bulk was cut into small disc (10 mm diameter) with a polished surface. Ag rod (99.99% purity, 2 mm in diameter, 2 mm in length) was placed with the disc into the high temperature contact instrument (OCA25HTV, Dataphysics, Germany), then was heated to 1120 $^{\circ}C$ for 10 min at a heating rate

of 8 $^{\circ}C/min$. The wetting angle data of molten Ag on the surface of Ti_2AlC were recorded by the software (SCA20).

1.3 Preparation of Ag/ Ti_2AlC composite

Powders of Ag (~10 μm , 99.99%) and Ti_2AlC (~20 μm , 99.2%) were weighed with a mass ratio of 9:1. Mixture powders were wet ball milled for 0.5 h with the medium of alcohol. Dried mixture were cold-pressed at 800 MPa into green body (15 mm in diameter, 3 mm in thickness). Samples were heated to 800 $^{\circ}C$ for 2 h in a tube furnace with a heating rate of 5 $^{\circ}C/min$.

1.4 Arc erosion experiment

Ag/TAC bulk was cut into the contacts (1.5 mm in thickness, 7 mm in diameter), then these contacts were welded with copper bases, finally were installed into a commercial contactor (CJX-50, China). The electric arc discharging experiment was carried out at the low voltage electrical apparatus test center of Shanghai Electrical Research Institute. The arc erosion resistance of Ag/TAC contacts were tested with the following experimental parameter (set according to the national standard GB14048.4-2010): AC-3 inductive load, 400 V voltage, 100 A circuit, 50 Hz coil frequency, 600 cycles/h operation frequency.

1.5 Characterizations

The phases were characterized by X-ray diffraction (XRD) (Bruker-AXS D8, Germany) with a scan rate of 10 ($^{\circ}$)/min. The morphologies of Ti_2AlC powder and Ag/10TAC composites were obtained by a field-emission scanning electron microscopy (FE-SEM) (Sirion 200, FEI/Philips, Netherlands), and their chemical component were tested and analyzed by the energy dispersed X-ray spectrometer (EDS) (AZtes X-MAX 80, Germany). The density of Ag/10TAC bulk was directly tested with the densimeter (DH-300, China, 0.001 g/cm^3 accuracy). The hardness of Ag/10TAC composite was measured by the Vickers microhardness tester (FM-700, Japan, 1 kg pressure, holding time of 5 s). The resistance of Ag/10TAC bulk was obtained by using the instrument (METRAHIT 27 I, Germany, 0.001 $m\Omega$ accuracy), and then its resistivity was calculated according to the four probe principle. Mass loss of the Ag/TAC contact after arc erosion was obtained by using the electronic balance (BT25S, Sartorius, Germany, 0.01 mg accuracy). Area loss of the contact after arc erosion was calculated with the metallographic microscope analysis software (LAS V4.8, Leica, Germany).

2 Results and discussion

2.1 High-purity Ti_2AlC powders

During the preparation of Ti_2AlC , Al first melts due to its low melting point and provides a liquid environment

for the subsequent reactions. Vaporization of Al at high temperature leads to the loss of Al in the raw materials. Therefore, appropriate increase of Al content is the premise to ensure the chemical reaction according to the stoichiometric ratio of raw powder. In Table 1, the content of TiC and Ti in the raw materials remains unchanged, excessive Al are added to the raw materials to study the effect of Al content on the purity of Ti₂AlC powder.

X-ray diffraction was carried out to identify the phase composition of the as-prepared Ti₂AlC powder. As shown in Fig. 1, peaks of Ti₂AlC were detected in all four samples, however, some impure phases like Ti₃AlC₂ and TiC were also detected in the final sample. Diffraction peak of the impure phase increases with the increase of the Al content in the raw materials. The purity of Ti₂AlC in the four samples are calculated according to the integral area of the main peak of the main phase^[24], as shown in Table 1. The result shows that the highest purity of Ti₂AlC powder reaches 77.5% (Sample S1) when the molar ratio of Al is 1.05 in the starting raw material.

Based on the Sample S1, the contents of Ti and Al in the raw materials remain unchanged, while the content of C reduces gradually. Four samples (Table 2) were designed to study the effect of C-deficiency in the raw material on the purity of Ti₂AlC powder. As shown in Fig. 2, XRD result shows that Ti₃AlC₂ and TiC phases are hardly detected in Sample S5, and the purity of Ti₂AlC powder was calculated as 99.2% (Table 2), indicating that the C-deficiency in the raw materials facilitates the generation of more Ti₂AlC phase. However, the further increase of C content in the raw material in turn leads to the

Table 1 Molar ratio of raw material powder TiC/Ti/xAl

Sample	Composition, x	Purity/%
S1	1.05	77.5
S2	1.10	57.9
S3	1.15	41.4
S4	1.20	31.5

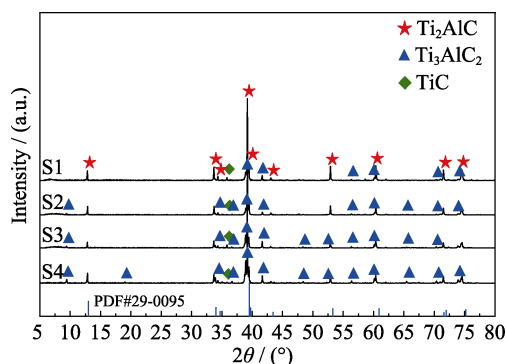


Fig. 1 XRD patterns of samples with different Al contents

Table 2 Molar ratio of raw material powder (2-x)TiC/xTi/1.05Al

Sample	Composition, x	Purity/%
S1	1.00	77.5
S5	1.05	99.2
S6	1.10	92.3
S7	1.15	91.7

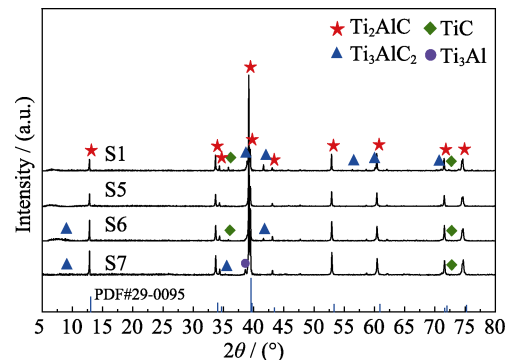


Fig. 2 XRD patterns of samples with different C contents

decomposition of Ti₂AlC into TiC and Ti-Al compound (Sample S6, S7). In addition, little Ti₃AlC₂ is also detected in the final samples. The purities of both Sample 6 and Sample 7 are less than 93%, which indicates that the severe shortage of C in the raw material deteriorates the purity of Ti₂AlC powder.

The morphologies of samples with different C contents are displayed in Fig. 3. The powders of Sample S1, S6 and S7 exhibit irregular shape and their surfaces were covered by many adherent particles with a small size of 0.2–0.8 μm (Fig. 3(a-b, e-h)). It is reported that the existence of two M layers in every two A layers leads to the formation of layered crystal structure of 211MAX phase^[11]. But the more C and excessive Al in raw material powders generate the impurity phase (TiC, Ti-Al compounds, *etc.*) in addition to the main phase Ti₂AlC, thus leading to the destruction of the crystal structure of Ti₂AlC and its poor morphology with nonstandard layered structure. In contrast, powder of Sample S5 shows standard lamellar structure of MAX without any adherent particles (Fig. 3(c, d)), indicating that the Ti₂AlC powder in Sample S5 have superior purity. This result presents that the slightly deficient C and a little more Al in raw materials do not produce impurity phase and the prepared Ti₂AlC have complete crystal structure, which is consistent with that of XRD result in Fig. 2.

Sintering temperature is another important factor for the phase transition and morphology evolution of the Ti₂AlC powder during the synthesizing process. Raw powder with the composition of 0.95TiC/1.05Ti/1.05Al is heated to different temperatures, as shown in Table 3. XRD result shows that the final powder contains a large

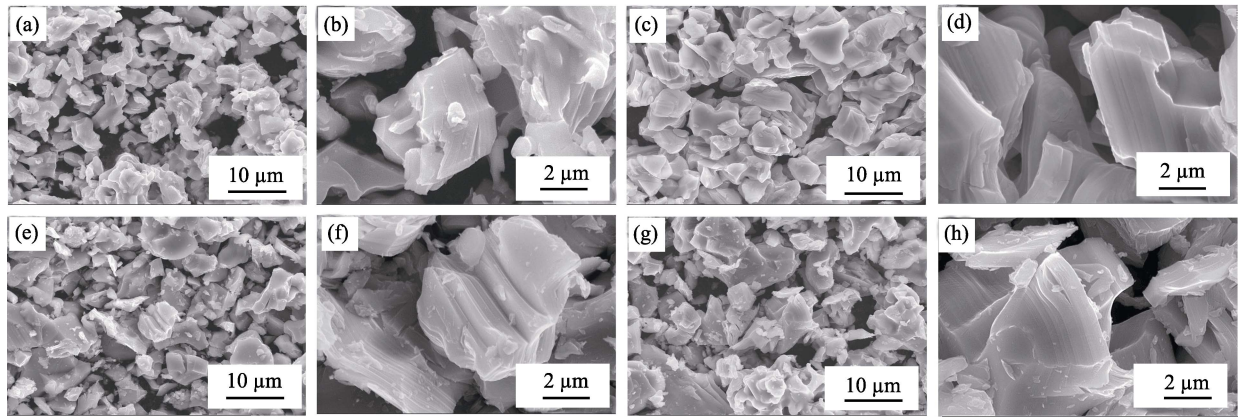


Fig. 3 SEM images of samples with different C contents (a, b) S1; (c, d) S5; (e, f) S6; (g, h) S7

amount of TiC and Ti-Al compounds due to the insufficient reaction when synthetic temperature is below 1400 °C (Fig. 4). Once the temperature exceeds 1500 °C, the synthesized Ti₂AlC phase largely decomposes into TiC and Ti-Al compounds, and merely produces little Ti₃AlC₂. At 1400 °C, the high-purity Ti₂AlC is stable, and no impure phase is detected.

In summary, appropriate increase of Al and slight reduction of C in the raw material facilitate the generation of Ti₂AlC phase in the final powder. But excessively low and high temperature hinder the purity improvement of Ti₂AlC powder. In order to obtain high-purity Ti₂AlC powder, the optimal synthesis process parameters is concluded as: a raw powder with composition of 0.95TiC/1.05Ti/1.05Al, sintered at 1400 °C for 1 h under pressureless condition.

Table 3 Preparation of Ti₂AlC powder from 0.95TiC/1.05Ti/1.05Al at different temperatures

Sample	Temperature/°C	Purity/%
S8	1200	16.3
S9	1300	35.7
S10	1400	99.2
S11	1500	81.8

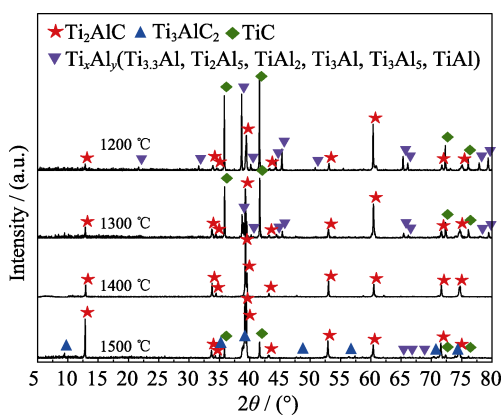


Fig. 4 XRD patterns of 0.95TiC/1.05Ti/1.05Al sintered at different temperatures

2.2 Wetting behavior of Ag/Ti₂AlC

In the process of heating, the contact angles and optical images of molten Ag on the Ti₂AlC substrate are simultaneously recorded, as shown in Fig. 5. The poor wettability of Ag and Ti₂AlC is observed at ~990 °C (a large contact angle of 154.6°). As temperature increases, Al atoms in Ti₂AlC outward diffuse into Ag matrix, leading to the reactive wetting and hence the decrease of contact angle. When temperature rises to 1130 °C, the contact angle stabilized at 14.1°, indicating that Ag well wets Ti₂AlC. In the preparation of Ag/Ti₂AlC composite, good wettability is beneficial to enhance the interface bonding between Ag and Ti₂AlC, and reduce the aggregation of Ti₂AlC particles. Good wettability also facilitates the viscosity improvement of the molten pool and the increases the resistance to splash of liquid Ag during the electric arc discharging.

2.3 Ag/Ti₂AlC composite

XRD result of Ag/10wt%Ti₂AlC (Ag/10TAC) composite is shown in Fig. 6. Except Ag and Ti₂AlC, no other impure phase were detected. The SEM image of Ag/10TAC (Fig. 6(b)) shows the uniform microstructure of the composite, and the good bonding of the Ti₂AlC with Ag matrix (Fig. 6(c)), which is attributed to the good wettability between the Ag and the Ti₂AlC. The basic properties

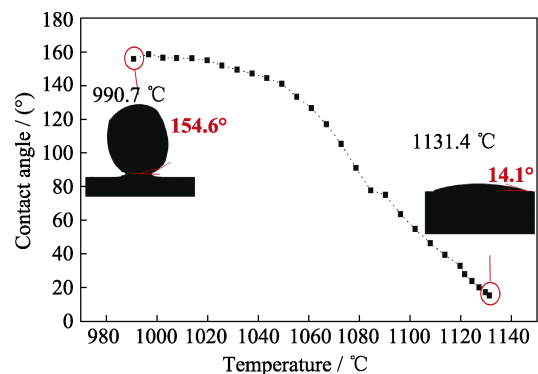


Fig. 5 Contact angles and optical images of Ag/Ti₂AlC in the process of heating

(density, relative density, hardness, resistivity) of Ag/10TAC composite are listed in Table 4. High relative density (~95.7%) and moderate hardness (96HV) provide good strength and machinability of the Ag/10TAC composite. Low resistivity (79.5 nΩ·m) facilitates the electric conducting of current and thus reduces contact resistivity and temperature rise of the Ag/10TAC contact.

2.4 Arc erosion resistance of Ag/10TAC

Arc erosion resistance is an important index to evaluate the application potential of an electrical contact material. As shown in Fig. 7, after 5610 electric arc discharging cycles, the shape of Ag/10TAC contact remains well (inset of Fig. 7(a)). The erosion area of eroded contact surface accounts for 88.6% of the total contact area,

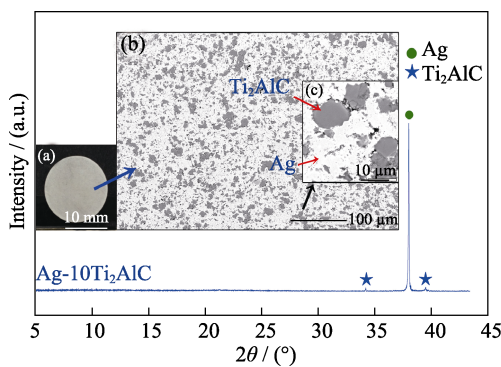


Fig. 6 XRD pattern of Ag/10TAC composite with insets showing (a) picture of the bulk, (b) microstructure of composite, and (c) the magnified SEM image

Table 4 Basic properties of Ag/Ti₂AlC composite

Density/ (g·cm ⁻³)	Relative density/%	Hardness, HV	Resistivity/ (nΩ·m)
8.692	95.7%	96	79.5

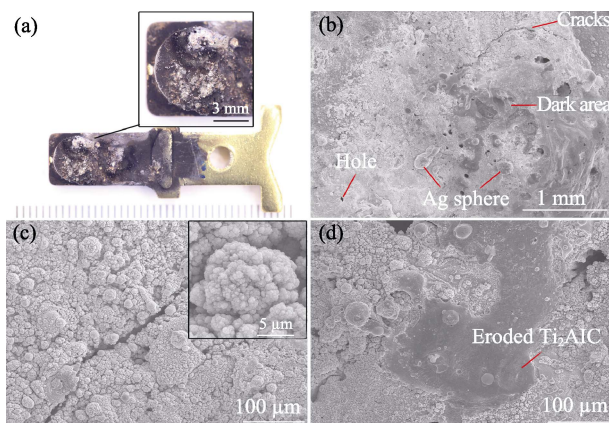


Fig. 7 (a) Optical image of the Ag/10TAC contact after 5610 arc discharging cycles, with magnified SEM image in the inset; (b) SEM image of the contact surface morphology; (c) Morphology of the Ag molten pool, with magnified SEM image of cauliflower-shaped Ag particles in the inset; (d) Magnified SEM image of the aggregated eroded Ti₂AlC (dark area in (b))

but the mass loss is only 4.4wt%, indicating that Ag/10TAC has strong resistance to arc erosion. Fine thermal and low resistivity of Ti₂AlC, good wettability between molten Ag and Ti₂AlC, and the interdiffusion between Ag and Ti₂AlC are responsible for the good contacting properties of the Ag/10TAC composite. After arc erosion, a few cracks, holes, and Ag spheres were observed on the surface of Ag/10TAC contact (Fig. 7(b)). The Ag matrix inside molten pool mainly exhibits cauliflower-shaped after arc erosion, as shown in inset of Fig. 7(c). In addition, some dark areas were observed on the contact surface (Fig. 7(b, d)), which mainly contains Ti, O, C, and little Al by EDS. The formation of these dark areas is attributed to the aggregation of the eroded Ti₂AlC, which has been partially destroyed by high-temperature oxidation during electric arc discharging.

3 Conclusions

Pressureless synthesis was employed to study the effect of composition and sintering temperature on the purity and morphology of Ti₂AlC powder. The structure and properties of the Ag/10TAC composite electrical contact material are investigated. Main conclusions are drawn as follows:

- 1) High-purity Ti₂AlC powder (99.2%) is synthesized at 1400 °C/h by using the raw powder with the starting composition of 0.95TiC/1.05Ti/1.05Al.
- 2) Contact angle decreases with temperature increasing, due to the reactive wetting between molten Ag and Ti₂AlC. Small contact angle (14°) is obtained at 1130 °C. Good wettability facilitates the preparation and improves the arc erosion resistance of Ag/Ti₂AlC.
- 3) Ag/10TAC composite exhibits uniform structure, high density, moderate hardness, low resistivity, and favorable arc erosion resistance.
- 4) Ti₂AlC has been proven experimentally to be a promising reinforcement for Ag-based electric contact material.

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高纯 Ti₂AlC 粉末的无压制备及其在 Ag 基电触头材料的应用

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摘要: Ag 基电触头是低压开关的“心脏”, 触头无 Cd 化一直困扰着人们, 寻找新型环保电触头材料是目前低压开关领域研究的重点。本研究从 Ag 基电触头增强相材料设计入手, 利用简单快速的无压技术合成了高纯 Ti₂AlC 粉末 (99.2%), 制备的 Ag/Ti₂AlC 复合电触头材料组织均匀、Ti₂AlC 颗粒与 Ag 基体结合紧密、相对密度高 (95.7%)、硬度适中 (96HV)、导电性好 (电阻率低至 79.5 nΩ·m)、抗电弧侵蚀性能优良 (5610 次电弧放电后触头质量损失仅为 4.4wt%)。Ag/Ti₂AlC 优良的结构和性能主要归因于 Ti₂AlC 本身的导电导热性能和 Ag/Ti₂AlC 之间的润湿性。该复合材料在进一步深入研究后, 有望大面积应用并替代传统电触头材料。

关键词: Ti₂AlC; 电触头; 金属陶瓷复合; 导电性; 润湿性; 抗电弧侵蚀

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