Ag-based Electrical Contact Material Reinforced by Ti₃AlC₂ Ceramic and Its Derivative Ti₃C₂Tx

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Abstract: Ag-based electrical contact plays a key role in low-voltage switches, and the “universal” contact of Ag/CdO has been phasing out because of its environmental toxicity. As a new kind of two-dimensional carbide material with good electrical conductivity and thermal conductivity, Ti₃C₂Tx, a representative of MXenes has showed exceptional potential in various fields, including being the reinforcement phase in electrical contact materials to substitute for the toxic CdO. In this work, we successfully prepared the Ag/Ti₃C₂Tx composite by powder metallurgy. Phase and microstructure of the Ti₃C₂Tx and Ti₃AlC₂ were characterized by XRD and SEM. The relevant properties of Ag/Ti₃C₂Tx composite, such as electrical resistivity, microhardness, machinability, tensile strength, and anti-arc erosion performance are investigated and compared with those of Ag/Ti₃AlC₂. The Ag/Ti₃C₂Tx has a resistivity of 30×10⁻³ μΩ m, 29% lower than Ag/Ti₃AlC₂ (42×10⁻³ μΩ m) and excellent machinability with intermediate microhardness (64 HV), showing broad application prospect as non-toxic electrical contact materials. The improved conductivity of Ag/Ti₃C₂Tx composite is mainly attributed to the metallicity of Ti₃C₂Tx itself, the microstructural features rendered by the deformability of Ti₃C₂Tx. However, the tensile strength (32.77 MPa) of Ag/Ti₃C₂Tx is inferior to that of Ag/Ti₃AlC₂ (145.52 MPa) due to the lack of Al-Ag interdiffusion. The anti-arc erosion performance of Ag/Ti₃C₂Tx is also unmatchable with Ag/Ti₃AlC₂ due to the absence of Al layer. Although the arc erosion resistance of Ag/Ti₃C₂Tx needs to be further improved, the significantly improved electrical conductivity makes it a potential substitute of current toxic Ag/CdO material. The results of this work provide an exploration direction for developing new environmentally friendly electrical contact material in the further.

Keywords: metal-ceramic composite; MAX phase ceramic; MXenes; electrical conductivity; mechanical properties; anti-arc erosion performance

As the critical component in low-voltage switching device, Ag-based electrical contacts are widely applied in contactor, breaker and relay, etc. The service life of these devices largely depends on the properties of the electrical contact material[1]. The conventional material “Ag/CdO” has long been preferred because of its outstanding contacting and arc extinction properties since the middle of last century. However, the toxicity of CdO causes serious pollution problems, restricting its applications[2]. In the past few decades, Cd-free electrical contact materials, such as Ag/SnO₂, Ag/ZnO, Ag/C, Ag/Ni, have been studied extensively[3-7]. These substitutes cannot yet emulate Ag/CdO in terms of temperature rise, contact resistivity, machinability, arc erosion resistance, etc. Therefore, environment-friendly alternative with properties matching up CdO is in high demand.

Over the past decades, MAX phase[8-10], combining excellent properties of metal and ceramic, has been widely investigated in various fields[11-16]. As a representative member of MAX family, Ti₃AlC₂ has been used to reinforce...
Ag matrix as the electrical contact material\cite{17-21}. However, the electrical resistivity of the Ag/Ti$_3$AlC$_2$ composite is not satisfactory, which is initially attributed to the inter-diffusion between Al layer and Ag matrix\cite{22}. 2011, Gogotsi and Barsoum\cite{23-24} jointly obtained a new kind of carbide material (Ti$_3$C$_2$Tx) with two-dimensional structure, coined as MXenes, were produced by selectively etching off Al atom planes from its parent Ti$_3$AlC$_2$. To date, Ti$_3$C$_2$Tx has received great attentions of many applications\cite{25-29}. In addition to large specific surface area, Ti$_3$C$_2$Tx has good electrical conductivity, thermal-conducting property, and hydrophilicity\cite{30}, and thus it is promising reinforcement for electrical conductive composite. In particular, Ti$_3$C$_2$Tx has demonstrated its potential as an additive in composites with polymers (PVA, PAM, PEI, PAN, etc.), ceramics (MoS$_2$, TiO$_2$, etc.) and carbon materials (CNT, MWCNT, CNFs, etc.)\cite{31}. Hence, the electric conductive Ti$_3$C$_2$Tx is expected to reinforce Ag matrix as a new electrical contact material.

In this study, the application of MXenes to electrical contact material is explored. Ti$_3$C$_2$Tx reinforcing Ag-based composite was prepared by powder metallurgy, and its overall properties, such as electrical resistivity, hardness, machinability, tensile strength, and anti-arc erosion were investigated and compared with those of Ag-based composite reinforced by Ti$_3$AlC$_2$ ceramic. The mechanism of properties difference of the both samples were also analyzed and concluded. The research results would provide significant data for the design and preparation of the new generation of environment-friendly silver-ceramic composite electrical contact materials in the future.

**1 Experimental**

Base materials for composites were Ag (99.9%, ~10 μm, Xinshengfeng, China) and Ti$_3$AlC$_2$ (99.0%, ~10 μm, preparing in our lab with TiC (99%, ~5 μm, Aladdin, China), Ti (99.9%, ~50 μm, Aladdin, China), Al (99.7%, ~50 μm, Zhongnuo xincai, China). Ti$_3$C$_2$Tx was obtained by immersing Ti$_3$AlC$_2$ (5 g) into hydrofluoric acid (HF) solution (100 mL, 40wt%) for 24 h under magnetic stirring (40 °C )\cite{32}. Ag/10wt%Ti$_3$C$_2$Tx, (Ag/Ti$_3$C$_2$Tx) and Ag/10wt%Ti$_3$AlC$_2$ (Ag/Ti$_3$AlC$_2$) mixtures were individually homogenized by ball milling for 0.5 h with a medium of ethyl alcohol (99.7% purity, Shanghai Titan Scientific Co. Ltd., China) according to the mass ratio. These two mixtures were subsequently cold-pressed into green bodies (15 mm in diameter, 2 mm in thickness) under 800 MPa, and then heat treated at 700 °C for 2 h in argon atmosphere.

Phase composition of the samples was characterized by X-ray diffraction (XRD, Bruker-AXS D8, Germany). The structure change of Ti$_3$AlC$_2$ and Ti$_3$C$_2$Tx powders were further characterized by Transmission Electron Microscopy (TEM) (FEI, Nova Nano 450, America). Vickers hardness of samples was measured by the four probe method (Metra HIT 27 I, Gossen Metrawatt, Germany). Microstructure and chemical compositions were characterized by a scanning electron microscope (SEM, FEI/Philips Sirion 2000, Netherlands), equipped with an energy dispersive spectrometer (EDS, Aztes X-MAX 80). The Ag/Ti$_3$C$_2$Tx and Ag/Ti$_3$AlC$_2$ bulk materials were processed into the dumbbell-shaped samples with length of 40.0 mm, width of 7.5 mm, middle part width of 4 mm and thickness of 2.0 mm. The tensile strength of both samples was tested at a universal test machine (AGS-X5kN, SHIMADZU, Japan) with a speed of 1 mm·min$^{-1}$. Finally, the Ti$_3$AlC$_2$-reinforcing and Ti$_3$C$_2$Tx-reinforcing Ag-based composite electrical contact were installed in commercial contactors and tested under the harsh conditions (400V/100A/AC3, GB14048.4-2010) at Low Voltage Switch Testing Center of Shanghai Electrical Appliance Research Institute.

**2 Results and discussion**

Fig. 1 shows the phase compositions and microstructures of raw powders (Ti$_3$AlC$_2$ and Ti$_3$C$_2$Tx). Ti$_3$AlC$_2$ was characterized by granular morphology with smooth surfaces (Fig. 1(a)), and Ti$_3$C$_2$Tx exhibited a multilayered morphology with the layer thickness of 0.15~0.37 μm (Fig. 1(b)). Fig. 1(b) obviously shows that the (002) diffraction peak of Ti$_3$C$_2$Tx is tilted towards low angle, which also confirms the expansion of Ti$_3$C$_2$Tx layer space.
Fig. 1 Phase and micro-morphologies of the reinforcing phase
(a) Ti₃AlC₂; (b) Ti₃C₂Tx

The microstructures and element distributions of Ag/Ti₃AlC₂ and Ag/Ti₃C₂Tx composites are displayed in Fig. 2. As shown in Fig. 2(a) and 2(c), both reinforcements (Ti₃AlC₂ and Ti₃C₂Tx) uniformly distribute in Ag matrices, Ti₃AlC₂ retains the granular morphology while Ti₃C₂Tx takes the stripe-shaped morphology. Fig. 2(b) and 2(d) displays the element distributions of Ag, Ti and Al in composites, which further confirms that Ti₃AlC₂ and Ti₃C₂Tx take different shapes in Ag matrices. Moreover, slight diffusion of Al with Ag is observed in Ag/Ti₃AlC₂ (Fig. 2(b)), while a few Al element is detected in Ag/Ti₃C₂Tx (Fig. 2(d)), which is consistent with the XRD and TEM results.

Ag/Ti₃C₂Tx composite is further observed at higher magnification SEM (Fig. 3(a)). It is obvious that the interface between Ti₃C₂Tx and Ag matrix is clear with no trace of cracks and holes, indicating a good physical bonding. However, Ti₃C₂Tx has large contact angle with Ag substrate during high-temperature wetting experiment (Fig. 3(b)), which confirms the absence of reactive wetting (i.e. chemical bonding) between them.

Fig. 2 Microstructure with SEM image and Element area distribution of composites
(a, b) Ag/Ti₃AlC₂; (c, d) Ag/Ti₃C₂Tx

Fig. 3 High-magnification SEM image of Ti₃C₂Tx in Ag matrix (a) and high-temperature wettability of Ti₃C₂Tx with Ag (b)

The insets in (b) show optical images of contact angle at different temperatures

Contact materials are usually manufactured into various shapes, necessitating excellent machinability. A typical
negative case is SnO$_2$ whose high hardness leads to the poor machinability of Ag/SnO$_2$, which hinders its substitute to CdO$^{[32]}$. Fig. 4 shows the Vickers hardness of Ag/Ti$_3$C$_2$T$_x$, Ag/Ti$_3$AlC$_2$, and pure Ag (for reference). Ag/Ti$_3$C$_2$T$_x$ possesses intermediate hardness (64 HV), and can be cut into different shapes, including rod, rivet, disc and square (the insert in Fig. 4). The good machinability originates from the 2D structure of Ti$_3$C$_2$T$_x$, in which weak van der Waals interaction exists between layers. In addition, contacts usually carry high current density in service, thus a low resistivity is a prerequisite for potential electrical contact materials. As shown in Fig. 4, the Ag/Ti$_3$C$_2$T$_x$ and Ag/Ti$_3$AlC$_2$ composites own low resistivity (16×10$^{-3}$ μΩ·m of Ag). In particular, the resistivity of Ag/Ti$_3$C$_2$T$_x$ (30×10$^{-3}$ μΩ·m) is 29% lower than that of Ag/Ti$_3$AlC$_2$ (42×10$^{-3}$ μΩ·m), which is very meaningful for the practice application.

The improved conductivity of Ag/Ti$_3$C$_2$T$_x$ can be explained from three aspects: higher conductivity of Ti$_3$C$_2$T$_x$ than that of Ti$_3$AlC$_2$, enhanced interface bonding between Ag and Ti$_3$C$_2$T$_x$, deformability of the stripe-shaped Ti$_3$C$_2$T$_x$ in Ag matrix.

Firstly, based on the first-principle band structure calculations, in Ti$_3$AlC$_2$, Ti3d state contributes the majority of the total densities of states (DOS) at Fermi level; removal of the Al layers from Ti$_3$AlC$_2$ results in the redistribution of Ti3d states from broken Ti-Al bonds into delocalized Ti-Ti metallic-like bonding states, leading to the increase of local DOS maximums at Fermi level$^{[33]}$. Thus, in MXene (Ti$_3$C$_2$T$_x$), the electron density of states near Fermi level (N(E$_F$)) is 1.9–3.2 times higher than that in the corresponding MAX (Ti$_3$AlC$_2$)$^{[34]}$. Secondly, EDS mapping indicates the existence of O and F elements, which may come from the functional groups (–F, –OH) of Ti$_3$C$_2$T$_x$ surface$^{[35]}$ (Fig. 5). Generally, the hydrophilicity of –F/OH functional groups is beneficial to the bonding between Ti$_3$C$_2$T$_x$ and metal matrix$^{[36]}$, which avoids the similar phenomenon of poor interface bonding between carbon nanotubes, fibers and metal matrix$^{[37]}$. In addition, the SEM observation also displays the tight bonding between Ti$_3$C$_2$T$_x$ and Ag matrix without obvious cracks and holes, as shown in Fig. 2(c) and Fig. 3(a). Hence, the uniform microstructure and good bonding of Ag/Ti$_3$C$_2$T$_x$ improved the conductivity.

Thirdly, as shown in Fig. 2(b, d), the microstructure of stripe-shaped Ti$_3$C$_2$T$_x$ is obviously different from that of granular Ti$_3$AlC$_2$ in Ag matrix. The 2D layered structure of Ti$_3$C$_2$T$_x$ facilitates its deformability during preparation. The Ti$_3$C$_2$T$_x$ was cold compacted into stripe-like Ti$_3$C$_2$T$_x$ (average thickness of ~3 microns), whereas Ti$_3$AlC$_2$ retains its original shape (average diameter of ~10 microns). In contrast with granular Ti$_3$AlC$_2$, stripe-shaped Ti$_3$C$_2$T$_x$ has smaller cross-sectional area perpendicular to the current direction, minimizing the scattering section for electrons and the resistance to the electron transmission. In summary, the excellent machinability and electrical conductivity makes Ag/Ti$_3$C$_2$T$_x$ a promising substitute for Ag/CdO.

The insert is pieces cut from Ag/Ti$_3$C$_2$T$_x$.

![Fig. 4 Machinability, electrical resistivity (blue bar) and Vickers hardness (red bar) of Ag/Ti$_3$C$_2$T$_x$ and Ag/Ti$_3$AlC$_2$, compared with those of Ag](image)

![Fig. 5 Micro-morphology and elements are distribution of Ag/Ti$_3$C$_2$T$_x$ composites at high-magnification SEM image](image)

However, as shown in Fig. 6, the maximum tensile strength of Ag/Ti$_3$C$_2$T$_x$ composite (32.77 MPa) is far less...
than that of Ag/Ti$_3$AlC$_2$ composite (145.52 MPa). The superior tensile strength of Ag/Ti$_3$AlC$_2$ composite derives from the interdiffusion between the active Al atomic layer with Ag matrix. On the contrary, the absence of Al layer leads to the weaker interface bonding strength between Ti$_3$C$_2$Tx and Ag matrix, which finally deteriorates the mechanical property of the entire Ag/Ti$_3$C$_2$Tx composite.

In order to further investigate the property of Ag/Ti$_3$C$_2$Tx, electrical arc discharging experiments were carried out on this contact surface under a harsh condition (AC-3, 100A, 400V, GB14048.4-2010). The Ag/Ti$_3$C$_2$Tx contact failed to make and break after 1233 arc discharging. The optical image shows that the shape of contact remains well with some dents and protuberances (Fig. 7(a, b)). The surface morphologies of Ag/Ti$_3$C$_2$Tx contact after arc erosion are subsequently exhibited in Fig. 7(c), complete edge and flat surface were further confirmed by SEM. Some Ag spheres, solidified Ag blocks, and small cracks were observed at high-magnification SEM image (Fig. 7(d)). Fig. 7(e-h) exhibit the microstructure and chemical composition of Ag/Ti$_3$C$_2$Tx contact surface. There are many irregular dark block surrounded by little bright particles (Fig. 7(e)). As shown in Fig. 7(f), area 1 (dark block) contains large amount of Ti, O, F with trace of Ag and Al, which may be attributed to the Ti-O-F mixture produced by electrical arc erosion to Ti$_3$C$_2$Tx. Area 2 (bright particles) mainly composes of Ag, F, and O. It is deduced that the Ag-O-F mixture were produced by the absorption of O$_2$ in liquid Ag and reaction with -F function group of Ti$_3$C$_2$Tx. Fig. 7(h) displays two types of spheroid particles at magnified SEM image. EDS analysis results show that both the particles contained vast N element, which indicated that these two particles compose of Ag-O-F-N.

The relative mass loss of Ag/Ti$_3$C$_2$Tx (54% after 1233 times arc discharging) is considerably more than that of Ag/Ti$_3$AlC$_2$ (0.82% after 3000 times arc discharging), which is also attributed to the absence of Al layer in Ti$_3$AlC$_2$. As analyzed previously, the lack of Al-Ag interdiffusion leads to the weak bonding strength of Ti$_3$C$_2$Tx with Ag, and thus decrease the mechanical property of composite, which accordingly impairs the resistance to electrical arc impact damage. In addition, the absence of Al-Ag interdiffusion also results in the poor wettability of Ti$_3$C$_2$Tx with Ag during electrical arc discharging and consequently decrease viscosity of molten pool, finally deteriorating the resistance to the material transfer of Ti$_3$C$_2$Tx and Ag under electrical arc high-temperature. Nonetheless, there is still space for the
further improvement of the arc erosion resistance of Ag/Ti$_3$C$_2$T$_x$ with superior electrical conductivity by the composition design, structure optimization, technique promotion in the following work.

3 Conclusions

In this work, Ag-based electrical contact materials, reinforced by Ti$_3$AlC$_2$ ceramic and its derivative (Ti$_3$C$_2$T$_x$), were successfully prepared by powder metallurgy. The microstructure, chemical composition, hardness, conductivity, machinability, mechanical property and erosion resistance of Ag/Ti$_3$AlC$_2$ and Ag/Ti$_3$C$_2$T$_x$ composites were investigated and compared. The main conclusions are as follows:

1) Stripe-like Ti$_3$C$_2$T$_x$ uniformly distributes in Ag/Ti$_3$C$_2$T$_x$ composite.

2) In contrast with Ag/Ti$_3$AlC$_2$, Ag/Ti$_3$C$_2$T$_x$ has lower resistivity (30×10$^{-3}$ μΩ·m), 29% lower than that of Ag/Ti$_3$AlC$_2$. The superior conductivity of Ag/Ti$_3$C$_2$T$_x$ results from the stronger metallicity of Ti$_3$C$_2$T$_x$, uniform microstructure, and smaller cross-sectional area of Ti$_3$C$_2$T$_x$ in Ag/Ti$_3$C$_2$T$_x$ composite.

3) The moderate hardness and excellent machinability of Ag/Ti$_3$C$_2$T$_x$ are also satisfactory for electrical contact materials.

4) The tensile strength (32.77 MPa) of Ag/Ti$_3$C$_2$T$_x$ composite is inferior to that of Ag/Ti$_3$AlC$_2$ (145.52 MPa) due to the lack of Al-Ag interdiffusion.

5) Ag/Ti$_3$C$_2$T$_x$ shows moderate arc erosion resistance with production of Ti-O-F, Ag-O-F, and Ag-O-F-N mixture, which is inferior to that of Ag/Ti$_3$AlC$_2$, and needs to be further improved.

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Ti₃AlC₂陶瓷及其衍生物 Ti₃C₂Tₓ 增强的 Ag 基电接触材料

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摘 要：银基电触头在低压开关领域扮演重要角色。作为一种具有良好导电导热性能的新型二维碳化物材料, MXene 家族典型代表材料 (Ti₃C₂Tₓ) 在多个领域显示出极大的应用潜力。Ti₃C₂Tₓ 有望作为一种新型环保银基电触头增强相材料。本研究采用粉末冶金法制备了 Ag/Ti₃C₂Tₓ 复合材料, 并通过 XRD 和 SEM 对 Ti₃C₂Tₓ 和 Ti₃AlC₂ 的物相和微观结构进行表征。同时研究了 Ti₃C₂Tₓ 增强 Ag 基复合材料的综合性能, 包括电阻率、显微硬度、机械加工性能、抗拉强度和抗电弧侵蚀性能, 并与 Ti₃AlC₂ 增强 Ag 基复合材料进行了比较。Ag/Ti₃C₂Tₓ 的电导率 (30×10⁻³ μΩ·m) 相对于 Ag/Ti₃AlC₂ (42×10⁻³ μΩ·m) 降低了 29%。由于硬度适中 (64 HV), Ag/Ti₃C₂Tₓ 具有良好的可加工性, 作为无毒电触头材料应用前景广阔。Ag/Ti₃C₂Tₓ 复合材料导电性能的提高主要归因于 Ti₃C₂Tₓ 本身优异的金属性以及由 Ti₃C₂Tₓ 微观结构特征所带来的可变形性。由于缺乏 Al-Ag 相互扩散, Ag/Ti₃C₂Tₓ 复合材料的拉伸强度 (32.77 MPa) 明显低于 Ag/Ti₃AlC₂ 复合材料 (145.52 MPa)。正因为 Al 层的缺失, Ag/Ti₃C₂Tₓ 的抗电弧侵蚀性能也低于 Ag/Ti₃AlC₂ 相媲美, 尽管 Ag/Ti₃C₂Tₓ 的抗电弧侵蚀性能有进一步提高, 但其优异的导电性使其有望替代当前有毒 Ag/CdO 材料。该研究结果为开发新型环保电触头材料提供了新的探索方向。

关 键 词：金属陶瓷复合材料; MAX 相陶瓷; MXene; 导电性; 力学性能; 抗电弧侵蚀性能

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