

Effect of La Content on Tribological Behavior of Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La Nanocomposites

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Abstract: Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites were fabricated by vacuum hot-pressing sintering and hot isostatic pressing. Effect of lanthanum (La) content on tribological behavior of Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites against GCr15 was evaluated using a wear and abrasion tester. In addition, influence of La content, normal force and rotational speed on tribological behavior of the nanocomposite was investigated to analyze their interaction mechanism. The mutual influence of La content, normal force and rotational speed was analyzed by orthogonality analysis, variance analysis and range analysis. The morphologies of worn surfaces have been observed and analyzed by scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS). Results demonstrate that La content plays an exclusively significant role in the friction and wear performance of the Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites. The wear mechanism of the nanocomposites with 0.05 wt% La is abrasive wear, delamination wear and oxidation wear. And wear mechanism of the nanocomposites with 0.1 wt% and 0.3 wt% La is adhesive wear and oxidation wear.

Key words: Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites; friction and wear; self-lubricating; wear mechanism; orthogonal analysis

Copper (Cu) matrix self-lubricating composites are widely used in many industrial applications, such as friction materials, bearings, bushes, brake pad/disc, collector shoe of maglev train, due to their good thermal and electrical conductivities, excellent tribological and mechanical properties, and ease of production^[1-4]. Graphite (C) and Ti₃SiC₂ are promising lubricants and reinforcements to fabricate Cu matrix composites with improved tribological performance and mechanical property owing to the lamellar structure of graphite and unique properties of Ti₃SiC₂ from metal and ceramics combined contribution^[5-8]. Multi-walled Carbon Nanotubes (MWCNTs) and Graphenes, one- and two-dimensional nanocarbons with unique thermal, electrical and mechanical properties, employed as reinforcements in composites, can improve their mechanical and tribological properties^[9-15]. For the rare-earth lanthanum (La) it has been reported that an appropriate

amount of La can promote grain refinement and grain-boundary strengthening by purification of harmful impurities in cast Cu or Cu-La alloys, thus improving their tensile strength, hardness and wearability^[16-21]. Nevertheless, the research on La reinforced metal matrix composites is still very inadequate; most of the existing literatures focus on the effect of La content on the particle characteristics and mechanical properties of Cu or Ti matrix composites^[22,23], whereas few literatures have been published to discuss the effect of La on the tribological behaviors of Cu matrix composites, especially for multi-phase reinforced Cu matrix composites.

In the present research, multi-phase reinforcements including La, Graphene, MWCNTs, Graphite, and Ti₃SiC₂ were used to fabricate Cu matrix nanocomposites by vacuum hot-pressing sintering and hot isostatic pressing. The effect of

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La on the friction and wear characteristics of the nanocomposites was explored using an orthogonal analytical method. An analysis was conducted to reveal the tribological mechanisms of the nanocomposites with different chemical components.

1 Experiment

Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites were prepared with the raw materials of rare-earth lanthanum La (0 wt%, 0.05 wt%, 0.1 wt%, 0.3 wt%), Graphene (0.2 wt%), MWCNTs (0.8 wt%), Graphite (0.3 wt%), Ti₃SiC₂ (10wt%) and electrolytic copper powder by a vacuum hot-pressing sintering and hot isostatic pressing method. Based on our previous research, Graphite and Ti₃SiC₂ were chosen as lubricant-components, and Graphene and MWCNTs were employed as reinforcements as well as lubricants to fabricate Cu matrix composites for the application of braking friction materials. After powder mixing and compacting of raw materials, composite powders were firstly sintered by the vacuum hot-pressing sintering method, i.e. heating the powder from room temperature to 950 °C and pressing it by a pressure of 20 MPa for 2 h, followed by a release of pressure and cooling inside the furnace to room temperature. Secondly, the composites were resintered by a hot isostatic pressing method, i.e. heating the presintered composites from room temperature to 650 °C and pressing them by a pressure of 0.6 MPa, subsequently pressing and heating to the final condition of 900 °C/100 MPa, keeping for 2 h, followed by rapid cooling (a cooling rate of 20 °C/min) inside the furnace to room temperature by introducing Ar gas as a protection medium.

The friction and wear experiments were taken on a M-2000A wear and abrasion tester by adjusting normal force and rotational speed to explore the friction and wear behavior of Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites under different working conditions as shown in Table 1 and Table 2. The counterparts in the experiments adopted GCr15 ball with a diameter of 42 mm due to its high hardness, good abrasion resistance and high contact fatigue performance. The morphologies of worn surfaces have been observed and analyzed by scanning electron microscope (SEM, JEOL

JSM-7001F) and energy dispersive X-ray spectroscopy (EDS) analysis.

2 Results and Discussion

2.1 Orthogonal analysis on tribological behavior of Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites

Orthogonality analysis was used to study the mutual influence of three factors including as La contents, normal force and rotational speed. In this way we can analyze the primary factor affecting friction and wear characteristics of Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites and get an ideal working condition. The testing results are obtained through variance analysis and range analysis as shown in Table 3.

2.1.1 Wear loss analysis

Table 4 is obtained for indicatrix of various factors based on an intuitive analysis for the experimental results. It demonstrates that La content has a greater impact on the wear loss than the other two factors of the normal force and rotational speed. It could be seen from Table 4 that, for the wear loss, the best level combination of various factors is A₂B₁C₄, i.e., when La content, normal force and rotational speed are 0.1 wt%, 350 N and 50 r/min, respectively.

Meanwhile, a larger range of the factor indicates a greater impact of the change in the factor level on the index whereas a smaller factor indicates a smaller impact. It could be seen from Table 4 that the ranges of various factors affecting the index is in the following sequence: R_A>R_B>R_C, indicating the dominative effect of La content on the wear loss compared to normal force and rotational speed. Moreover, we took a variance analysis of three factors to analyze the primary factor affecting the wear loss of the Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites. When $F_y = MS_y / MS_e > F(1-\alpha)$

Table 1 Factors and levels of orthogonality analysis test

Factor	Level 1	Level 2	Level 3	Level 4
A La content/wt%	0.05	0.1	0.3	0
B Normal force/N	350	400	450	500
C Speed/r·min ⁻¹	200	150	100	50
D Error term	1	2	3	4

Table 2 Orthogonality analysis test-L16 (4⁴)

Test No.	A La content/ wt%	B Normal force/N	C Speed/ r·min ⁻¹	D Error.	Test No.	A La content/ wt%	B Normal force/N	C Speed/ r·min ⁻¹	D Error.
1	0.05	350	200	1	9	0.3	350	100	4
2	0.05	400	150	2	10	0.3	400	50	3
3	0.05	450	100	3	11	0.3	450	200	2
4	0.05	500	50	4	12	0.3	500	150	1
5	0.1	350	150	3	13	0	350	50	2
6	0.1	400	200	4	14	0	400	100	1
7	0.1	450	50	1	15	0	450	150	4
8	0.1	500	100	2	16	0	500	200	3

Table 3 Conditions and results of orthogonality analysis test- L16 (4⁴)

Test No.	A La content/wt%	B Normal force/N	C Speed/r·min ⁻¹	D Error	Wear loss/g	Friction coefficient
1	0.05	350	200	1	0.0141	0.226
2	0.05	400	150	2	0.0136	0.228
3	0.05	450	100	3	0.0138	0.239
4	0.05	500	50	4	0.0115	0.239
5	0.1	350	150	3	0.0064	0.245
6	0.1	400	200	4	0.0129	0.240
7	0.1	450	50	1	0.0105	0.272
8	0.1	500	100	2	0.0164	0.244
9	0.3	350	100	4	0.0087	0.237
10	0.3	400	50	3	0.0082	0.243
11	0.3	450	200	2	0.0214	0.233
12	0.3	500	150	1	0.0160	0.237
13	0	350	50	2	0.0029	0.387
14	0	400	100	1	0.0046	0.362
15	0	450	150	4	0.2150	0.412
16	0	500	200	3	0.1783	0.406

Table 4 Factors-wear loss analysis result

Factor allocation	A La content/wt%	B Normal force/N	C Speed/r·min ⁻¹	D Error
Mean value K_1	0.0133	0.00802	0.05668	0.01130
Mean value K_2	0.0116	0.00983	0.06275	0.01358
Mean value K_3	0.0136	0.06518	0.01088	0.05168
Mean value K_4	0.1002	0.05555	0.00828	0.06203
Range	0.0886	0.05716	0.05447	0.05073

Table 5 Work conditions-wear loss variance analysis

Factor allocation	Sum of deviation square	Freedom	F ratio	F critical value	Significance
La content/wt%	0.4701	3	49.404	($\alpha=0.05$) 7.23	Yes
Normal force/N	0.0082	3	0.8587	($\alpha=0.10$) 2.61	-
Rotational speed/r·min ⁻¹	0.0076	3	0.7977	($\alpha=0.10$) 2.61	-

(F_y, F_e), it is considered that the factor is significant at the significance level α , i.e., effect of the factor is not negligible^[24]. The test table F ratio obtained from calculation is shown in Table 5. It shows only the factor of La content has a significant effect on the wear loss of nanocomposites according to the variance analyses.

2.1.2 Friction coefficient analysis

Table 6 is obtained for indicatrix of various factors based on

an intuitive analysis for the experimental results. It demonstrates that La content has a primary impact on the friction coefficient, followed by the normal force and rotational speed. It could be seen from Table 6 that, for the friction coefficient, the best level combination of various factors is $A_1B_2C_3$, i.e., when La content, normal force and rotational speed are 0.05 wt%, 400 N and 100 r/min, respectively. The range of various factors affecting the friction coefficient is similar

Table 6 Factors-friction coefficient analysis result

Factor allocation	A La content/wt%	B Normal force/N	C Speed/r·min ⁻¹	D Error
Mean value K_1	0.2330	0.2738	0.2763	0.27425
Mean value K_2	0.2503	0.2683	0.2805	0.2730
Mean value K_3	0.2375	0.2890	0.2705	0.2833
Mean value K_4	0.3918	0.2815	0.2853	0.2820
Range	0.1588	0.0207	0.0148	0.0103

Table 7 Work conditions-friction coefficient variance analysis

Factor allocation	Sum of deviation square	Freedom	F ratio	F critical value	Significance
La content/wt%	0.0692	3	117.14	($\alpha=0.005$) 7.23	Yes
Normal force/N	3.267×10^{-5}	3	0.0553	($\alpha=0.10$) 2.61	-
Speed/r·min ⁻¹	1.570×10^{-5}	3	0.0266	($\alpha=0.10$) 2.61	-

to wear loss as the following sequence: $R_A > R_B > R_C$. Additionally, as shown in Table 7, the calculated F ratio (117.14) is larger than the F critical value (7.23) at the significance level α (0.005) when the factor of La content is considered, suggesting the exclusively significant effect of La content on the friction coefficient of nanocomposites rather than the other two factors of the normal force and the rotational speed.

2.2 Effect of La content on friction and wear characteristics

According to the orthogonal analyses shown in Table 4, the wear loss of nanocomposites without La addition (K_4) is obviously greater than that of the nanocomposites with presence of La (K_1 , K_2 and K_3). However, no significant difference is observed from the wear loss of nanocomposites with increasing La content from 0.05 wt% to 0.3 wt%. The friction coefficient of the nanocomposites without La addition (K_4) is obviously greater than those of others with presence of La (K_1 , K_2 and K_3) as shown in Table 6, while, no significant difference is observed with increasing La content from 0.05 wt% to 0.3 wt%. Obviously, the addition of La can decrease both wear loss and friction coefficient as an indicator of improving abrasive resistance of the nanocomposites. This improvement could be attributed to the effects of both solid solution strengthening and grain refinement strengthening introduced by La during sintering of the Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites, which have been discussed in other literatures^[22,23]. However, the aggregation of La is observed after powder mixing, and porosity of the nanocomposites is slightly increased with the increment of La content, which would weaken the increasingly solid solution strengthening effect caused by the increment of La content.

2.3 Effect of normal force on friction and wear characteristics

Under dry friction and wear conditions, the effects of normal force or applied load and rotational speed on the wear rate of composites can be represented by the Archard model of wear calculation as Eq. (1).

$$W = KP V / H \quad (1)$$

Where, W is the wear rate, K is friction coefficient, P is load, V is rotational speed, and H is hardness of the composites.

It can be seen from Table 4 that the wear loss firstly rises slightly when the load is increased to 400 N (K_2); an obvious increment is observed when the load is increased to 450 N (K_3); whereas a decrease is observed when the load is further increased to 500 N (K_4). At the beginning, since the surface of the nanocomposites is coarse and uneven to some extent, the grinding balls contact the micro-bulge on the surface of the

nanocomposites directly, which is cut and pulled to form rigid abrasive grains under the effect of frictional force. Partial abrasive grains are detached and partial abrasive grains retain between the friction pair to serve as the third abrasive grains. A lower load results in a slighter wear rate according to Eq.(1) and a slight wear loss caused by a relatively few quantity of detached abrasive grains. With the increment of applied load, the stress of contacting surface rises and the plastic deformation increases, even a friction node occurs^[25]. In addition, the dual lubricant components of Ti₃SiC₂ and graphite existing in the nanocomposites act as a lubricant film at the interface of friction pair, whereas, this lubricant film could be perforated or destroyed under a high load, thus providing ineffective protection for the surface of the composites and accelerating wear. The wear loss increases noticeably with an increasing load, which could be resulted from severe abrasive wear affected by third abrasive grains along with adhesive wear affected by shearing of friction node, as well as the failure of the lubricant film. A similar tendency of the friction coefficient as the wear loss affected by the normal force is observed in Table 6, although the difference is not so significant. The lubricant film caused by the lubricants of Ti₃SiC₂ and graphite at the friction interface plays a significant role in the characteristic of friction coefficient.

2.4 Effect of rotational speed on friction and wear characteristics

The friction and wear behavior the of composites under different rotational speed is similar to that under different loads. As shown in Table 4. An obvious increment of wear loss is observed when the rotational speed is increased from 100 r/min (K_2) to 150 r/min (K_3), while a decrease is observed when the rotational speed is further increased to 200 r/min (K_4). Under a lower rotational speed (~ 100 r/min), the shear stress between the friction pair is small. The dual lubricant components of Ti₃SiC₂ and graphite existing in the composites are tripped in the lamellar form and overlaid onto the matrix surface to form a lubricant film during friction process. The velocity of the lubricant film formation is larger than or equal to its failure velocity, thus effectively protecting the surface of the composites and alleviating wear. Whereas, with increasing the rotational speed (150 r/min), the shear stress between the friction pair increases^[24]. Under a larger shear stress, the agglomerated reinforcement is detached from Cu matrix nanocomposite surface in the granular form. The granular abrasive grains enhance the plough-effect of the friction pair, resulting in destroying and failure of the lubricant film.

Additionally, more heat is generated at the friction interface in the case of a higher rotational speed, followed by a rise of temperature and softening of the composites at the contacting interface. The plastic deformation of the composites at the interface increases and the resistance to shear strength decreases. The worn surface is inclined to initiate fatigue cracks after circles, resulting in the detachment of abrasive grains from the nanocomposites, which serve as the third abrasive grains to accelerate wear of the surface^[26-31]. A

similar tendency of the friction coefficient as the wear loss affected by the rotational speed is observed from Table 6 as analyzed above.

2.5 Mechanism of friction and wear characteristics

Since the factor of the normal force has a greater impact on both wear loss and friction coefficient than the rotational speed, three test conditions are selected to investigate the effect of La content on the mechanism of friction and wear for SEM and EDS characterization of worn surfaces and debris as

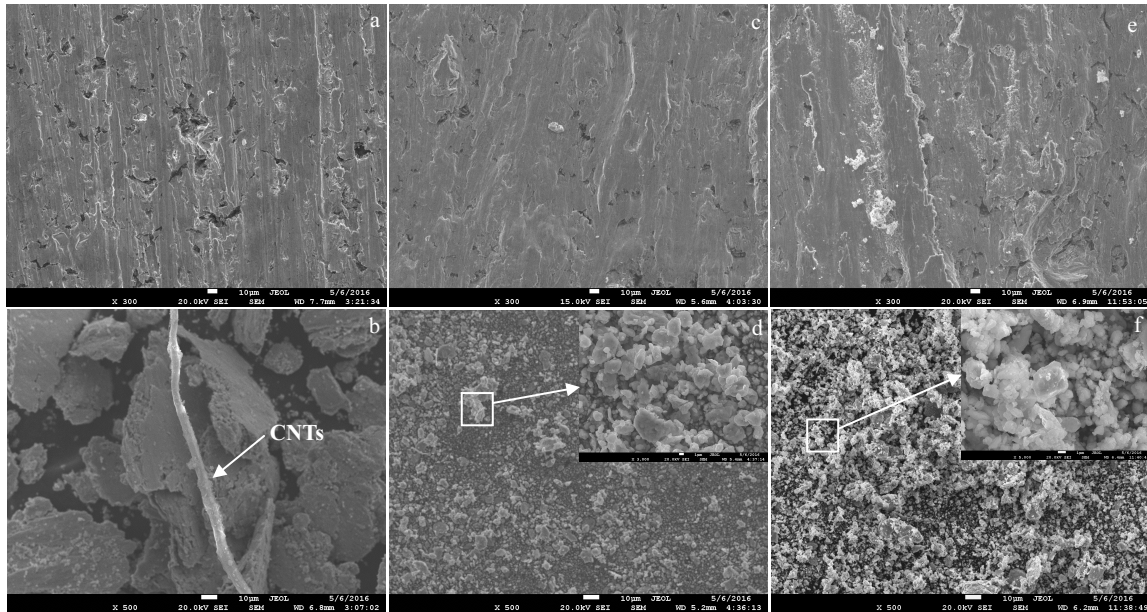


Fig.1 SEM micrographs of worn surfaces and debris of Cu matrix nanocomposites with different La content: (a, b) 0.05 wt%, (c, d) 0.1 wt%, and (e, f) 0.3 wt%

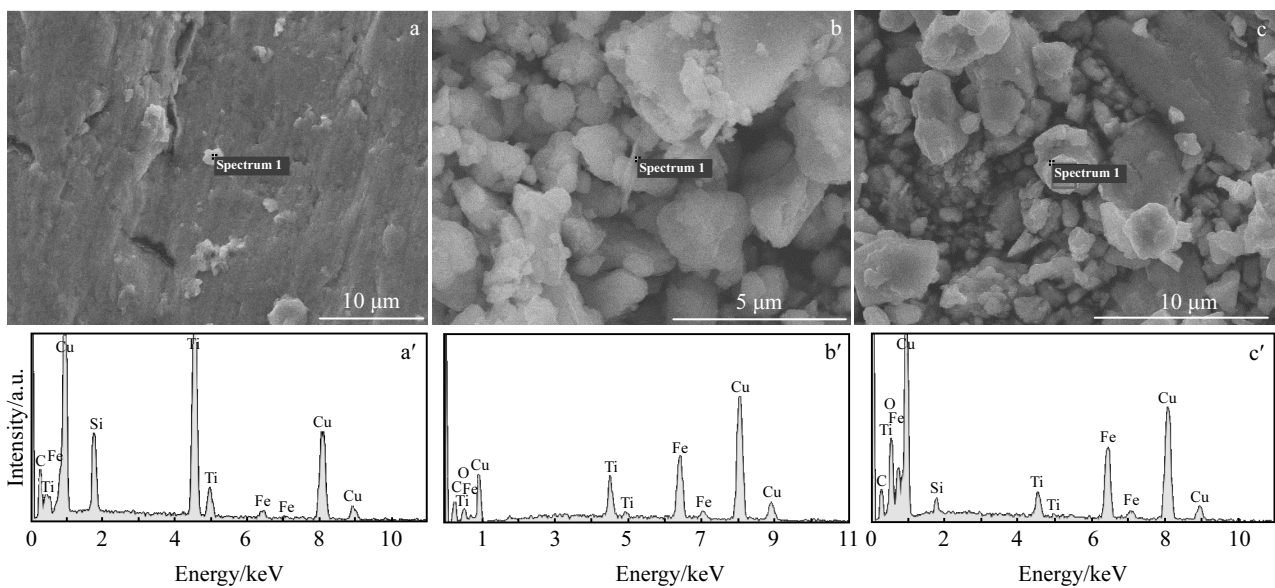


Fig.2 SEM morphologies (a~c) EDS spectra (a'~c') of worn debris of Cu matrix nanocomposites with different La content: (a, a') 0.05 wt%, (b, b') 0.1 wt%, (c, c') 0.3 wt%

described in Table 3 (test No. 1, 5 and 9). Fig.1 shows SEM micrographs of worn surfaces and debris of the Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites with different La contents. A small quantity of debris is observed on the worn surface when the La content is 0.05 wt%, and furrows are distributed along the direction of friction, which are the characteristic of abrasive wear. The morphology of the lamellar debris shown in Fig.1b indicates the possibility of delamination wear. The EDS result of the debris shown in Fig.2a suggests the possibility of components as third abrasive particles primarily including Ti₃SiC₂ or TiC decomposed from Ti₃SiC₂, graphite or CNTs or graphene, adhesion or aggregated particles of the Cu matrix, oxidized particles of metals as well. Meanwhile, oxidation wear is easy to occur as adsorption of oxygen on the surface is much easier in working conditions. When La content is 0.1 wt%, few debris is observed on the worn surface and the furrows disappear. The morphology of the debris shown in Fig.1d is no longer lamellar, but almost round edging and granular particles, suggesting the characteristic of adhesive wear. When La content is 0.3 wt%, the amount of debris on the worn surface increases, and the debris is fine and granular particles as shown in Fig.1f. The friction node can be formed by shearing or transferring at the interface of friction due to the pressure and movement of the contacting surface, and some fine debris stick together to form a particle of larger size, suggesting an obvious characteristic of adhesive wear^[27,29]. The EDS results of the debris shown in Fig.2b and 2c with different La contents indicate similar components of the particles, suggesting the possibility of components as third abrasive particles primarily including adhesion or aggregated particles of the Cu matrix and grinding ball of GC15, oxidized particles of metals, Ti₃SiC₂ or TiC decomposed from Ti₃SiC₂, graphite or CNTs or graphene as well. Consequently, in this research La can improve the tribological performance of the Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites. The wear mechanism of the Cu matrix nanocomposites with La content of 0.05 wt% is abrasive wear, delamination wear, as well as oxidation wear, and the wear mechanism of the nanocomposites with La content of 0.1 wt% and 0.3 wt% is adhesive wear, and oxidation wear as well.

3 Conclusions

1) During the process of dry friction and wear of the Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La nanocomposites, La content is the primary factor affecting the tribological behavior of composites, followed by the normal force and the rotational speed respectively.

2) La can improve tribological performance of the nanocomposites. The results of range analysis show that La content has a significant influence on the friction and wear behavior among the three factors. The ideal working condition is: La content=0.1 wt%, $F=350$ N, $n=50$ r/min.

3) Under the best working condition of normal force, the wear mechanism of lower La content of 0.05 wt% is abrasive wear, delamination wear and oxidation wear, while the wear mechanism of higher La content of 0.1 wt% and 0.3 wt% is adhesive wear and oxidation wear.

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镧对 Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La 纳米复合材料摩擦学性能的影响

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摘要: 研究采用真空热压及热等静压方法制备 Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La 纳米复合材料, 采用摩擦磨损试验机研究对磨材料为 GCr15 时, 镧含量对 Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La 纳米复合材料的摩擦学性能的影响。研究了镧含量、正应力及旋转速度对纳米复合材料摩擦学行为的影响并揭示其相互作用机理, 采用正交试验分析、方差分析及极差分析法来分析镧含量、正应力及旋转速度的相互作用。采用扫描电镜和能谱仪观察并分析磨损表面及磨削的形态及成分组分。结果表明, 镧对纳米复合材料的摩擦磨损性能起到首要作用, 当镧的质量分数为 0.05% 时, 复合材料的磨损机理为磨粒磨损、剥层磨损和氧化磨损, 而当镧的质量分数为 0.1% 和 0.3% 时, 复合材料的磨损机理为粘着磨损和氧化磨损。

关键词: Cu/Ti₃SiC₂/C/MWCNTs/Graphene/La 纳米复合材料; 摩擦磨损; 自润滑; 磨损机理; 正交分析

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