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RESEARCH

Tribology of Ti_2/Cr_2 and $(TiCr)_2AlC_x$ MAX Phase Ceramics

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A B S T R A C T

The MAX phase ceramics namely Ti_2AlC_x, Cr_2AlC_x, and $(TiCr)_2AlC_x$ were examined under dry sliding conditions against stainless steel ball at room temperature and 5 N applied normal load using a Ball-on-disk tribometer. At 5 N, 100 m sliding distance and 20 cm/s sliding velocity, Cr_2AlC_x, and $(TiCr)_2AlC_x$ ceramic samples exhibited low specific wear rate (3.2×10^{-7} cm^3/Nm and 4.2×10^{-7} cm^3/Nm), while the coefficients of friction were 0.31 and 0.10, respectively. At 5 N, 100 m and increasing velocity from 5 cm/s to 20 cm/s, Ti_2AlC_x sample the coefficients of friction decreases from 0.25 to 0.14. The specific wear rate of Ti_2AlC_x sample was 6.7×10^{-7} cm^3/N m at 5 N, 100 m and 5 m/s, and decreases with increasing velocity (3.3×10^{-7} cm^3/N m at 20 m/s). The correlations between observed tribological properties and tribo-film characteristics were discussed.

1. INTRODUCTION

MAX phase ceramics are a fascinating class of materials that possess a unique mix of properties, including good electrical and thermal conductivity like metals, as well as high-temperature stability and Oxidation resistance like ceramics [1]. Among the MAX phase ceramics, Titanium/Chromium-based and (Titanium-Chromium) Aluminium Carbide (Ti/Cr) and combined $(TiCr)_2AlC_x$ have received much attention as a result of exceptional mechanical characteristics and potential applications in various fields [2].

The synthesis of MAX phase ceramics, that is Ti_2AlC_x, Cr_2AlC_x and $(TiCr)_2AlC_x$, involves several methods aimed at achieving dense and high-performance materials. Solid-state reaction is a conventional approach where powders of constituent elements are mixed in the desired stoichiometric ratios and heated at high temperatures to facilitate diffusion and reaction. Alternatively, spark plasma sintering is a rapid consolidation technique combining pulsed DC current and pressure to rapidly sinter the combination of powder, resulting in dense and fine-grained ceramics. Additionally, Hot Pressing (HP) and Hot Isostatic Pressing (HIP) are widely used methods to achieve densification under elevated temperatures and pressure. HP applies uniaxial pressure to the powder, while HIP utilizes isotropic pressure in an inert gas medium to achieve full densification and eliminate porosity. Pseudo-Hot Isostatic Synthesis (PHIP) modifies the HIP process by applying pressure uniaxially through a flexible container. These methods offer varying advantages in terms of control over microstructure and mechanical properties, allowing researchers to tailor the MAX phase ceramics for specific applications [3-5].

Ti_2AlC_x, Cr_2AlC_x, and $(TiCr)_2AlC_x$ are specific MAX phases that showed remarkable mechanical and thermal characteristics, making they promising candidates for numerous engineering applications. In particular, significantly higher melting points oxidation resistance, and ability to retain strength at elevated temperatures to make them useable in extreme environments. These mechanical characteristics MAX phase ceramics can vary significantly depending on their composition, microstructure, and synthesis route. For instance, Ti_3AlC_2 MAX phase ceramics exhibited a flexural strength ranging from 900 MPa to 310 - 427 MPa. Similarly, Ti_2AlC ceramics demonstrated a Vickers hardness of 3.5×10^3 to 5.7×10^3 MPa and flexural strength of 384 MPa [6-9]. The mechanical properties can also be affected by the occurrence of secondary phases and grain size distribution [10]. These variations in properties highlight the importance of optimizing the synthesis and processing parameters for specific applications.

The wear performance of these MAX phase materials, especially Ti_3SiC_2 and Ti_2AlC, demonstrates their potential as solid lubricants [11]. The presence of lubricious MAX phases like Ti_3SiC_2 in the microstructure can reduce friction and wear during sliding contact. The layered crystal structure of MAX phases allows for easy shear of individual layers, providing a self-lubricating effect [12-15]. The formation of a low-friction tribo-film during sliding can contribute to the solid lubrication behaviour. The relatively low coefficients of friction observed in some MAX phases, like Ti_3SiC_2, at low applied loads indicate the presence of inherent lubrication, which is highly desirable in applications where reduced friction and wear are crucial [16]. These findings highlight the potential of MAX phase materials as effective solid lubricants in various engineering applications.

The work of Gupta et al. [17,18] stands out as the most significant in recent research addressing the high-temperature tribology of MAX phase materials. Gupta and colleagues investigated the tribological behaviour of certain MAX phases against a Ni-based alloy at both ambient and high temperatures. Their major focus was on determining the effect of tribo-oxidation products on lubricity, with a special emphasis on disc material. While their research gave useful information, it created a vacuum in the understanding of microstructural change during tribological interactions. To address this constraint, the current work will explore the tribological capabilities of certain MAX phase composites under ambient temperatures.

The selection of tribological test parameters and geometry in this inquiry has been conducted meticulously so as to consistency use our earlier work at ambient conditions [15,17,18]. This critical decision aims to explore the existence of similar lubrication mechanisms at elevated temperatures compared to those observed under ambient conditions [19,20]. This careful selection of the test temperature allows for a thorough comprehension of the underlying tribological phenomena at elevated temperatures while mitigating the confounding influence of oxidative wear. The combination of chromium oxide (Cr_2O_3) and titanium dioxide (TiO_2) in coatings has been studied for its potential in enhancing wear and corrosion resistance can exhibit improved tribological properties, reducing wear and friction on the grey cast iron surface. [21] The research on the application of plasma-sprayed Cr_2O_3-40% TiO_2 coatings on brake discs, aiming to enhance tribological properties. Leveraging the excellent wear-resistant and hardness characteristics of chromium oxide and titanium dioxide, the study employs plasma spraying for its ability to produce dense coatings. The coated brake discs are expected to exhibit reduced friction, enhanced wear resistance, and improved durability compared to uncoated counterparts [22]. Examining the impact of distinct coatings on braking performance. The first investigation explores the effects of Cr_2O_3-$2TiO_2$ coating, while the second study focuses on $55TiO_2$-Cr_2O_3 coating. In both cases, the researchers likely assess tribological properties, including wear resistance and friction characteristics, to understand the coatings' influence on braking efficiency. These studies collectively contribute valuable insights into the optimization of brake materials and coatings, underscoring their importance in enhancing safety and overall braking system performance [23,24].

The current study aims to considerably contribute to the understanding of tribological properties of MAX phase materials using a similar strategy as Gupta and colleagues and filling gaps in their work. This study examines the wear and friction behaviour of different MAX phases as they slide across a stainless-steel ball at ambient temperature. Because stainless steel does not oxidise or wear, the results are excellent. This study's results will have major significance for possible applications in harsh operating circumstances due to the clarification of intrinsic lubrication processes and the function of microstructure evolution under diverse counterface materials. Wear resistance is a significant aspect impacting the practical practicality of these MAX phases in diverse applications.

2. EXPERIMENTAL DETAILS

2.1 Materials

In this study, the fabricated and characterized MAX phase ceramics are Ti_2AlC_x, Cr_2AlC_x, and $(TiCr)_2AlC_x$. The starting powders, titanium (Ti), chromium (Cr), aluminium (Al), and graphite (C) of particle size 44 µm, 40-50 µm, 41.6 µm, 75 µm were precisely weighed according to the stoichiometric ratios derived from the reactions. To ensure a homogenous mixture, we employed a horizontal revolving mixing mill with ZrO_2 balls and ethanol as the processing media, maintaining a powder-to-ball ratio of 1:3 for efficient mixing. Subsequently, the powder blend was dried at 100 °C for 2 h to remove the ethanol and ensure uniformity. The purity, particle size and source of the starting powders used to make the Ti_2AlC_x and Cr_2AlC_x and $(TiCr)_2AlC_x$ MAX phase ceramics are as follows:

$$2Ti + Al + {}_{X}C \rightarrow Ti_2AlC_x \quad (1)$$

$$2Cr + Al + {}_{X}C \rightarrow Cr_2AlC_x \quad (2)$$

$$Ti + Cr + Al + {}_{X}C \rightarrow (TiCr)_2AlC_x \quad (3)$$

2.2 Fabrication method

For the fabrication process, we utilized uniaxial pressing and reactive hot pressing (RHP). After mixing the dried powder blend with 2 wt. % of paraffin wax as a binding agent and hexane as a mixing medium, we uniaxially pressed the powders in EN24 steel dies, applying an appropriate pressure of 500 kg/cm^2 according to the sample's diameter. In order to accomplish the desired material properties and densification, we performed RHP at 1300 °C for 60 min in a lab-designed hot press under a flowing argon atmosphere. During RHP, temperature cycles were employed, gradually heating the samples to 1300 °C and then cooling them to room temperature at controlled rates.

Following the fabrication, we processed the MAX phase ceramics for analysis. The samples were cut using a low-speed cutting machine with a diamond blade and cold-mounted. We conducted utilising grinding and polishing SiC abrasive papers with different grit sizes, followed by polishing with 0.25 µm to 3 µm diamond suspension to obtain a specular reflection surface.

2.3 Wear testing

In the present work, the dry sliding wear behaviour of Ti_2AlC_x, Cr_2AlC_x, and $(TiCr)_2AlC_x$ MAX phases were investigated using Ball-on disk tribometer according to ASTM G 133-05 standard we evaluated the wear behaviour and the mechanics of MAX phase ceramics using a ball-on-disk tribometer under controlled conditions. The diameter of the ball (counter surface) is 3 mm. Our comprehensive experimental technique offers insightful information about the wear performance of these MAX phase ceramics, offering essential knowledge for potential good wear engineering applications.

Every experiment was performed using 30 mm diameter samples subjected to varying sliding velocity. A constant 5 N load and 100 m sliding distances were maintained for all the wear tests. Sliding velocities of 5 cm/s, 10 cm/s, 15 cm/s, and 20 cm/s were used during wear tests. Additionally, we examined wear behaviour by conducting tests at track radii of 5 mm, 6 mm, 7 mm, and 8 mm. To assess the wear performance, the length of wear tracks was precisely measured after each test.

2.4 Microhardness testing

Material hardness is often assessed by indentation with indenters. Measurements of Vickers hardness have been performed on the samples using a load of 500 g. The measurements were performed using an HSV-20 model hardness tester from Shimadzu Co. in Kyoto, Japan.

2.5 Scanning electron microscopy

Scanning Electron Microscope (SEM) photos were taken to examine the worn surfaces and determine the wear processes in each MAX phase. The findings of these experiments give crucial information on the wear resistance and tribological properties. The MAX phases of Ti_2AlCx, Cr_2AlCx, and $(TiCr)_2AlCx$ allow us to grasp their prospective applicability for typical engineering applications, which ranges from 50 to 200 MPa.

3. RESULTS AND DISCUSSION

3.1 Effect of MAX phases on specific wear rate

The following equation was used to compute the particular specific wear rate (W_r) of the MAX phases:

$$W_r = \frac{V_w}{F_n \times S} \tag{4}$$

where V_w is the volume of removed worn material in cm^3, F_n is the applied load in N and S is the sliding distance in m. The hardness of the steel ball was found to be 1000HV (Vickers hardness value) and diameter of 3 mm.

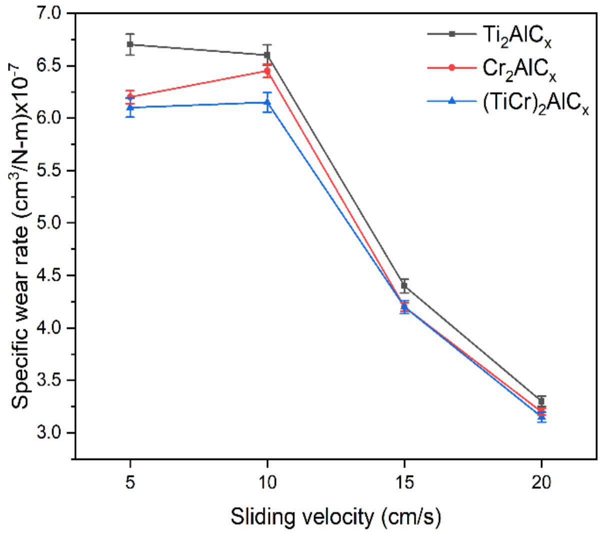


Fig. 1. Specific wear rate of MAX phase ceramics.

From Table 1 and Fig. 1 depicts that the specific wear rate (W_r) values obtained for the three materials were within the range of 3.1 ×10^{-7} cm^3/Nm to 6.7 × 10^{-7} cm^3/Nm. Interestingly, $(TiCr)_2AlC_x$ demonstrated the lowest W_r among the materials tested, indicating higher resistance to wear in comparison to Ti_2AlC_x and Cr_2AlC_x. The exceptional wear resistance of $(TiCr)_2AlCx$ can be because of its unique combination of mechanical properties. The presence of titanium (Ti) and chromium (Cr) whose hardness were 290HV and 750HV in the material's composition enhances its hardness and wear resistance.

Table 1. CoF and $\boldsymbol{W_r}$ of MAX phase ceramics.

Material	Sliding velocity (cm/s)	Coefficient of Friction (CoF)	Specific Wear rate × 10 $^{-7}$ (cm^3/Nm)
Ti_2AlC_x with steel Ball	5	0.25 ± 0.01	6.7 ±0.10
	10	0.23 ± 0.02	6.6 ±0.12
	15	0.18 ± 0.01	4.4 ±0.11
	20	0.14 ± 0.09	3.3 ±0.14
Cr_2AlC_x with steel Ball	5	0.38 ± 0.07	6.2 ±0.13
	10	0.36 ± 0.08	6.4 ±0.11
	15	0.38 ± 0.05	4.2±0.12
	20	0.31 ± 0.05	3.2 ±0.13
$(TiCr)_2AlC_x$ with steel Ball	5	0.18 ± 0.04	7.2 ±0.10
	10	0.14 ± 0.10	7.4 ±0.10
	15	0.12 ± 0.08	5.3 ±0.12
	20	0.10 ± 0.01	4.2±0.12

The TiC phase formation within the structure further strengthens the material, reducing wear-related deformation and abrasion. The combination of mechanical properties and microstructural characteristics makes $(TiCr)_2AlC_x$ highly resistant to wear under various sliding velocities as compared to Ti_2AlC_x, Cr_2AlC_x.

In contrast, Ti_2AlC_x displayed moderate wear resistance, showcasing a slightly higher W_r than $(TiCr)_2AlC_x$. The wear behaviour of Ti_2AlC_x can be attributed to its composition, which lacks the beneficial chromium content found in $(TiCr)_2AlC_x$. Similarly, Cr_2AlC_x showed a higher SWR compared to $(TiCr)_2AlC_x$, mainly due to the absence of titanium, which limits its wear resistance potential.

3.2 Effect of MAX phases on coefficient of friction

The CoF values obtained for Ti_2AlC_x, Cr_2AlC_x, and $(TiCr)_2AlC_x$ were in the range of 0.10 to 0.30 depicted in Fig. 2 and listed in Table 1. Across the sliding velocity range of 5 to 20 cm/s, for sliding velocities between 4 to 10 cm/s, the CoF decreased slightly and then slightly decreased. However, as the sliding velocity increased beyond 10 cm/s, the CoF decreased further, indicating a different frictional behaviour at higher sliding velocities. Beyond the velocity of 10 cm/s, the CoF experienced a more pronounced decrease. At greater sliding velocities, ploughing and mild wear processes are more common, which might explain this behaviour. As the sliding velocity increases materials may experience less time for plastic deformation, as a result, smoother and more continuous sliding motion, leading to lower friction.

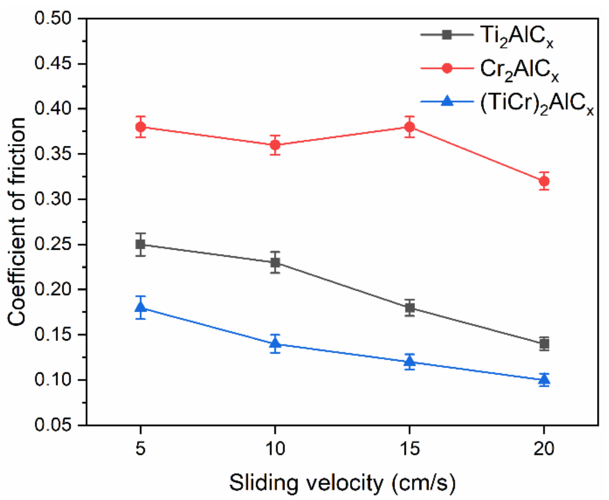


Fig. 2. Co-efficient of friction of MAX phase ceramics.

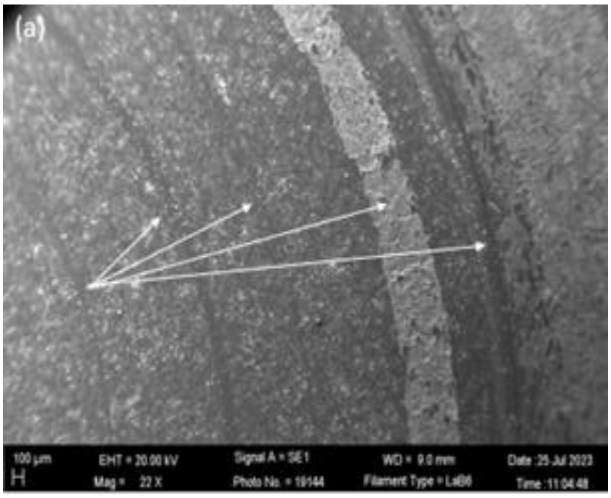
The coefficient of friction measures the resistance encountered between two surfaces in contact when one surface is shifted the other. In the present work, (TiCr)2AlCx material showed better frictional resistance than other materials. This suggests that (TiCr)2AlCx has a high resistance to sliding, which is often associated with higher friction, and sticking phenomena.

3.3 Effect of MAX phases on microhardness

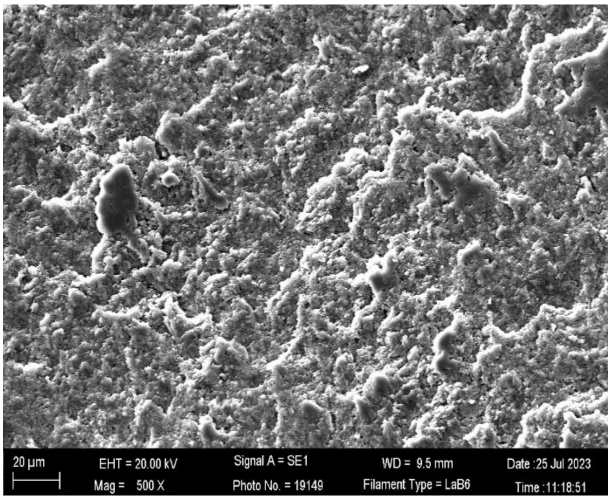
The microhardness obtained for Ti_2AlCx, Cr_2AlCx, and $(TiCr)_2AlCx$ were 6.2±0.5 GPa, 7.4 ± 0.8 GPa, and 8.4 ± 0.7 GPa, respectively. The microhardness of $(TiCr)_2AlCx$ is greater than the other MAX phases and it the most resistant to indentation and localized deformation. Cr_2AlCx follows closely in terms of microhardness, while Ti_2AlCx shows a slightly lower value compared to remaining materials. Comparing the microhardness values, $(TiCr)_2AlCx$ stands out as the hardest material, showcasing the highest resistance to indentation and possessing excellent localized deformation resistance [25]. This enhanced microhardness of $(TiCr)_2AlCx$ can be attributed to its unique composition, which likely contributes to stronger interatomic bonds and enhanced structural stability.

3.4 Wear track and worn surface morphology

Fig. 3(a, b) shows the tribo-surfaces of a Ti_2AlC_x ceramic sample under SEM after tribological testing with a 5 N load at 15 cm/s in ambient temperature. At lower magnification (20 X), the photomicrograph shown in Fig. 3a showed visible traces of wear on the shape of material removal and surface deformation. The wear tracks at a load of 5 N were observed at track radii, specifically at 5 mm, 6 mm, 7 mm, and 8 mm from the centre of the sample at a sliding velocity of 5 cm/s, 10 cm/s, 15 cm/s, 20 cm/s. The measured wear track lengths were 0.43 mm, 0.80 mm, 0.53 mm, 2 mm respectively. The microstructure within these wear tracks exhibited signs of plastic deformation, surface scratching, and abrasive wear. the presence of particles from wear and grooves along the track indicated the severity a process of wear. The overall trend of decreasing friction coefficient and specific wear rate (Figs. 1 and 2), with test time and speed can be explained by the fact that tiny particle fragments separated during the test might operate as a transfer film (15 cm/s and 7 mm track; Fig. 3), increasing wear resistance.



(a)

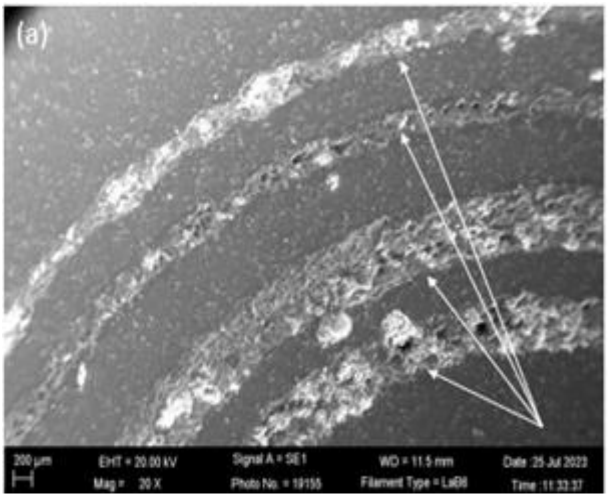


(b)

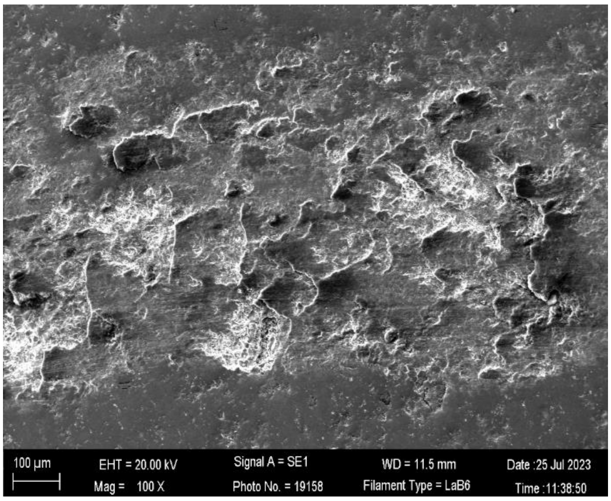
Fig. 3. Wear micrographs of Ti_2AlC_x at 20 X (a) and (b)100 X magnification.

At higher magnification (100 X) seen in Fig. 3b (contact conditions: wear track of 8 mm), allowed for a more detailed examination of the wear tracks. Observed within the were little cracks wear track, which indicated the initiation of microstructural damage due to wear-induced stresses. Additionally, spalling of material along the wear track indicated the occurrence of adhesive wear.

Fig. 4(a, b) shows the tribo-surfaces of a Cr_2AlC_x ceramic coupon under SEM after tribological testing with a 5 N load at 15 cm/s in ambient temperature. At lower magnification (20 X), the SEM image given in Fig. 4a displayed evident wear tracks at loads of 5 N specifically at 5 mm, 6 mm, 7 mm, and 8mm from the centre. the wear test was conducted at sliding velocities of 5 cm/s, 10 cm/s, 15cm/s, and 20cm/s. It was measured wear track lengths were 0.53 mm, 0.48 mm, 0.32 mm, and 0.16 mm respectively. Similar to Ti_2AlC_x, the wear tracks in Cr_2AlC_x exhibited plastic deformation, surface scratching, and the presence of wear debris. The severity of the wear was apparent from the wear-induced material removal and damage along the wear track.



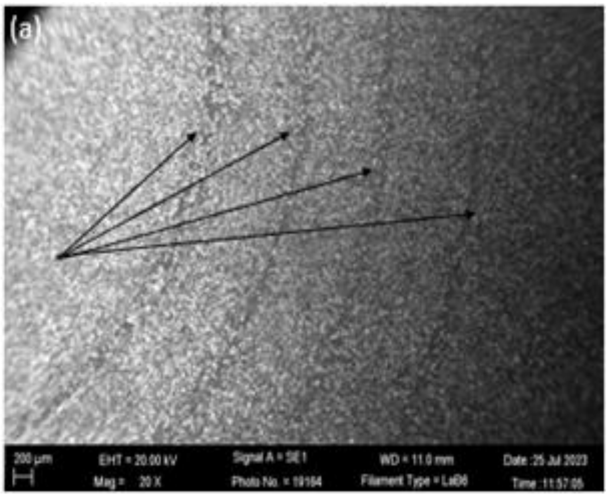
(a)



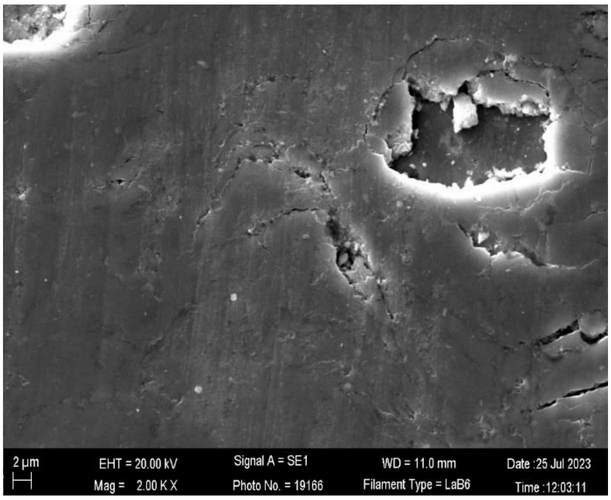
(b)

Fig. 4. Wear micrographs of Cr_2AlC_x (a) 20 X magnification, (b) 100 X magnification.

Examining the wear tracks at higher magnification (100 X) viewed in Fig. 4b (contact conditions: wear track of 8 mm). provided insights into the microstructural changes caused by wear. Microcracks and the worn surface within the wear track were more clearly visible which indicates the deformation caused by the wear. The distinct microstructural features of Cr_2AlC_x led to varying wear compared to Ti_2AlC_x, this is attributable to differences in crystal structure and hardness.



(a)



(b)

Fig. 5. Wear micrographs of $(TiCr)_2AlC_x$ (a) 20 X magnification, (b) 100 X magnification.

Fig. 5(a, b) shows the tribo-surfaces of a $(TiCr)_2AlC_x$ ceramic coupon under SEM after tribological testing with a 5 N load at 15 cm/s in ambient temperature. At lower magnification (20 X) depicted in Fig. 5a, images from SEM of the $(TiCr)_2AlC_x$ wear samples showed wear tracks at a load of 5 N and sliding velocity of 5 cm/s, 10 cm/s, 15 cm/s and 20 cm/s and at track radii of 5 mm, 6 mm, 7 mm, and 8 mm. The measurement of wear track lengths was 0.51 mm, 0.24 mm, 0.20 mm and 0.18 mm respectively. The wear tracks exhibited a combination of plastic deformation, surface scratching, and wear debris, comparable to the other two MAX phase ceramics. However, because of the existence of Ti and Cr in the composition.
Analysing the wear tracks at 100 X magnification illustrated in Fig. 5b allowed for a more detailed understanding of the microstructural changes induced by wear. The presence of microcracks and fractures within the wear track indicated the initiation of wear-induced damage. The formation of wear particles and grooves suggested the taking place abrasive wear, which is influenced by the hardness and strength of the material.

Ti_2AlC_x exhibited moderate wear resistance due to its combination of strength and hardness, but it was susceptible to adhesive wear and plastic deformation. Cr_2AlC_x displayed a different wear behaviour, characterized by abrasive wear and delamination. $(TiCr)_2AlC_x$, being a composite of Ti_2AlC_x and Cr_2AlC_x, showed a combination of wear mechanisms observed in both MAX phases, due to the presence of carbide particles, with the potential for better wear resistance.

4. CONCLUSIONS

The study investigated the Wear behaviour of Ti_2AlC_x, Cr_2AlC_x, and $(TiCr)_2AlC_x$ MAX phase ceramics. These materials exhibited varying wear resistance due to differences in their composition and microstructure, with $(TiCr)_2AlC_x$ showing superior wear resistance due to the presence of a combination of materials. The results are based on a single wear test performed.

- Ti_2AlC_x demonstrated moderate wear resistance, while Cr_2AlC_x exhibited relatively higher wear rates, emphasizing the importance of composition in determining wear performance.
- The coefficient of friction (CoF) was influenced by sliding velocity. The CoF decreased as sliding velocity increased beyond 10 cm/s, indicating changing frictional behaviour at higher speeds.
- Microscopic analysis of worn surfaces revealed wear tracks with plastic deformation, surface scratching, and wear debris. Being present microcracks indicated the initiation of wear-induced damage.
- The comparison between Ti_2AlC_x and Cr_2AlC_x demonstrates the importance of material composition (microstructure) in determining wear performance. Ti_2AlC_x is reported to have moderate wear resistance, while Cr_2AlC_x exhibits relatively higher wear rates. This highlights the role of material microstructure in influencing wear characteristics.

- The potential reason for decrease in the amount of wear as sliding speed increases, at higher sliding speeds, there may be a greater likelihood of the formation of protective layers on the sliding surfaces. These layers could include oxides or other compounds that act as barriers, reducing direct contact between the sliding materials and thus minimizing wear.

- Ti_2AlC may exhibit moderate wear resistance. The wear performance is influenced by the combination of the metallic behavior of titanium, the hardness of carbides, and the layered structure. Ti_2AlC's wear behavior may be favorable in certain applications, but it might not have the highest wear resistance among the three compositions.

- The low wear rate of Cr_2AlC_x compared to Ti_2AlC_x, despite its higher coefficient of friction, can be attributed to the favorable combination of material properties inherent to Cr_2AlC_x. The higher hardness of chromium, the formation of a protective oxide layer, and potential self-lubricating properties contribute to enhanced wear resistance. The microstructure, surface finish, and thermal stability of Cr_2AlC_x may also play pivotal roles in reducing wear rates, even under conditions of higher friction. The specific composition of Cr_2AlC_x appears to be optimized for wear performance, creating a material that effectively minimizes wear through a combination of mechanical strength, chemical stability, and tribological interactions at the sliding interface.

- The findings give insightful information on the wear characteristics of these ceramics, with $(TiCr)_2AlC_x$ standing out as an excellent potential candidate for engineering applications where wear resistance is crucial, especially in high-stress and high-speed conditions. Future research could focus on further optimizing the characteristics of $(TiCr)_2AlC_x$ for specific industrial uses to maximize its potential in demanding engineering scenarios.

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