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Abstract: A new class of ceramic matrix composites based on Cr₂AlC MAX-phase containing 5 and 10 wt.% of SiC fibers was developed in this investigation. The Cr₂AlC/SiC composites were performed through two consecutive steps: i) synthesis of the pure Cr₂AlC phase from its elemental constituents by reactive method under argon atmosphere at 1400 °C and particle size refinement, and ii) processing of Cr₂AlC powder and SiC fibers followed by densification using the field assisted sintering technology/spark plasma sintering. Cr₂AlC/SiC composites presented high density (98.6 %) with an excellent dispersion of the fibers within the matrix and a strong interaction matrix/fiber. Tribological behavior of the developed composites was studied under dry conditions to reveal the role played by the SiC fibers. Incorporation of the SiC fibers within the Cr₂AlC matrix reduced the friction coefficient up to 20 %, while the wear resistance was increased up to 80 %.

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Novel Cr₂AlC MAX-phase/SiC fiber composites: Synthesis, processing and tribological response

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Abstract

A new class of ceramic matrix composites based on Cr₂AlC MAX-phase containing 5 and 10 wt.% of SiC fibers was developed in this investigation. The Cr₂AlC/SiC composites were performed through two consecutive steps: i) synthesis of the pure Cr₂AlC phase from its elemental constituents by reactive method under argon atmosphere at 1400 °C and particle size refinement, and ii) processing of Cr₂AlC powder and SiC fibers followed by densification using the field assisted sintering technology/spark plasma sintering. Cr₂AlC/SiC composites presented high density (98.6 %) with an excellent dispersion of the fibers within the matrix and a strong interaction matrix/fiber. Tribological behavior of the developed composites was studied under dry conditions to reveal the role played by the SiC fibers. Incorporation of the SiC fibers within the Cr₂AlC matrix reduced the friction coefficient up to 20 %, while the wear resistance was increased up to 80 %.

1. Introduction

Nowadays, there is a strong demand to develop new functional materials that can operate under working conditions of high temperature, harsh environment, large wear rate and high stress in strategic fields such as energy, transportation, aerospace and communication. Under these conditions, large degradation occurs in the components limiting their durability and reliability - i.e. air-foil bearings, cylinders, gears, gas turbine seals, protection systems and rotational parts in engines. Metallic and/or superalloy compounds are the most used materials due to their excellent mechanical response [1], although they are limited by their tribological and mechanical capabilities at high temperature [2][3]. On the other hand, advanced ceramic materials such as SiC, Si₃N₄, Al₂O₃ or ZrB₂, have been typically used under high temperature, harsh environments and high wear conditions, but their inherent low fracture toughness and poor damage resistance have reduced their use in mechanical applications. To overcome

these restrictions, these advanced ceramics have been typically reinforced with particles, platelets, whiskers, and chopped and continuous fibers, leading to ceramic matrix composites (CMCs) [4]. The best mechanical results were commonly attained by adding continuous SiC fibers to a SiC matrix [5][6], although the complex processing routes for getting these CMCs, and the difficulty and high costs to machine intricate components have restricted their wide spread use at industrial scale. Consequently, a quantitative leap in material technology is mandatory for the next generation of functional materials. During the last years, a new family of compounds, called MAX phases, has strongly arose [7][8].

MAX phases are ternary nanolayered compounds with general formula of $M_{n+1}AX_n$, where M corresponds to an early transition metal, A is a A-group element (IIIA or IVA element), X is C and/or N, and n is typically equal to 1, 2 or 3 [9]. The interest of these phases (more than 70 different compounds have been reported [10]) resides on the unique combination of their properties, bridging the gap between ceramics and metals [11]. In general, MAX phases are lightweight, elastically stiff, good thermal conductors, and resistant to oxidation and chemical attack at high temperatures, as carbides and nitrides ceramics; while they are soft, easily machinable, thermal shock resistant and damage tolerant as metals. This singular response is linked to their crystal structure (space group P63/mmc): the M-X bond has a mixed covalent/ionic character, whereas the M-A bond is metallic. Nevertheless, there is a lack of comprehensive information about the properties of many MAX phases, which could be associated with the novelty of these compounds – properties of the firstly synthesized MAX phase were reported in 1996 by Barsoum et al. [12] - and the difficulty to obtain them. Consolidation of pure and highly dense bulk MAX phase materials is a challenge in itself, as second phases – mainly carbides and intermetallic compounds – are commonly observed in the final microstructure [13]. Typically, synthesis from the elemental constituents and sintering process are simultaneously carried out as both processes require high temperature. Nevertheless, a previous synthesis process to obtain firstly the pure MAX phase followed by the densification of the milled powder leads to a finer control of purity, microstructure and density [14]. Besides, sintering techniques assisted by pressure, such as hot pressing (HP) and hot isostatic pressing (HIP), or by current and pressure, as field assisted sintering technology/spark plasma sintering (FAST/SPS), are required to fully densify the MAX phase bulk materials [13].

Among all the MAX phases, Cr_2AlC is particularly attractive due to its combination of properties at room and high temperature [15], the excellent oxidation and corrosion resistances and the self-healing ability [16]. Regarding the mechanical properties, Cr_2AlC presents low hardness (3.5 – 5.5 GPa), high Young's modulus (288 GPa), intermediate fracture toughness ($5.2 \text{ MPa}\cdot\text{m}^{1/2}$), good flexural (555 GPa) and

compression strength (1160 MPa) [17][15]. Mechanical properties are retained up to 900 °C, presenting brittle-to-ductile transition temperature in the range of 800 – 900 °C [17]. Furthermore, Cr₂AlC bulk materials are good electrical ($2.3 \cdot 10^6 \Omega^{-1} \text{m}^{-1}$) and thermal conductors ($22 \text{ W} \cdot \text{m}^{-1} \text{K}^{-1}$) [18], high thermal shock resistant, and present a thermal expansion coefficient of $12 \cdot 10^{-6} \text{ K}^{-1}$, which is closed to that of the superalloys – potential applications in the field of ceramics/metals joining and protective coatings - [19]. They show excellent oxidation resistance at high temperature ($1.1 \cdot 10^{-11}$ and $3.0 \cdot 10^{-9} \text{ kg}^2 \text{m}^{-4} \text{s}^{-1}$ at 1000 °C and 1300 °C, respectively) and corrosion resistance against molten Na₂SO₄ at 900-1000 °C [20][21]. Recently, it has been reported that Cr₂AlC exhibits self –crack healing ability in the temperature range 900 – 1200 °C due to the filling of the cracks by oxides ($\alpha\text{-Al}_2\text{O}_3$ and $(\text{Cr}, \text{Al})_2\text{O}_3$) well adhering to the crack faces [16]. Concerning the tribological response of Cr₂AlC materials, limited number of works has been published [22][23][24], despite the promising results reported for this compound and other MAX phases, mainly Ti₃SiC₂. Cr₂AlC - and in general all the MAX phases - show intermediate friction coefficient (μ) and wear rate (W_R) at room and high temperature. W_R and μ values between $10^{-3} - 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \text{m}^{-1}$ and 0.5-1.0, respectively, were attained under dry conditions. This good response is caused by the formation of a protective and lubricious tribofilm during the sliding process, although it is a non-continuous layer triggering a loud metallic noisy tribological response and non-steady friction [24]. Tribological behavior of Cr₂AlC was enhanced through the incorporation of different contents of silver particles, which is the only measured Cr₂AlC composite investigated regarding its tribological behavior [25][26]. Friction coefficient and wear rate decreased under dry conditions from 0.64 and 0.003 $\text{mg} \cdot \text{s}^{-1}$ to 0.5 and 0.002 $\text{mg} \cdot \text{s}^{-1}$, respectively, with the incorporation of 20 vol.% Ag. Unfortunately, the lack of more tribological information about other Cr₂AlC composites restricts the prediction of the effect of second phases on this property.

MAX phase composites have been almost exclusively developed in Ti₃SiC₂ matrices, and just silver and Al₂O₃ particles have been incorporated into Cr₂AlC materials. TiC and SiC particles are the common second phases incorporated within Ti₃SiC₂, as these carbides are the typically reactants/intermediate products during the synthesis of this phase. Thus, their addition in higher concentrations to the MAX stoichiometry promotes the formation of MAX phase containing carbides. For example, the reaction between Ti, SiC and C leads to the formation of Ti₃SiC₂ containing 30 vol. % SiC, whereas reaction of Ti, SiC and TiC produces Ti₃SiC₂ containing 30 vol. % TiC [27]. The same approach has been also performed for Cr₂AlC composites, where Cr₂AlC/Al₂O₃ was obtained through the reaction of appropriated amounts of Cr₃C₂, Cr, Al and Cr₂O₃ [28]. Regarding MAX composites containing fibers, to the best of our knowledge, only three works have been reported [29][30][31], where commercial Ti₃AlC₂, Ti₃SiC₂ and

Ti₃AlC₂ were mixed with SiC fibers to analyze the matrix/fiber interface. Ti₂AlC powders reacted with the SiC fibers resulting in the formation of intermetallic compounds and carbides at the interface, whereas Ti₃SiC₂ and Ti₃AlC₂ presented no apparent reaction with the fibers.

The aim of the present work is to develop for the first time Cr₂AlC composites containing SiC fibers. Herein, we proposed a synthesis route to obtain pure Cr₂AlC phase and an easy mixing process with the SiC fibers, followed by the densification of the composites using FAST/SPS. The microstructure, degree of fiber dispersion and matrix-fiber interface were characterized, and the mechanical and tribological properties were evaluated.

2. Experimental procedure

The first step to consolidate the Cr₂AlC/SiC-fiber composites was to synthesize the pure Cr₂AlC phase. Chromium (Cr, 85.5 μm, 99.0 % pure), aluminum (Al, 9.1 μm, 99.5 % pure) and graphite (C, 7 – 11 μm, 99.0 % pure) powders (all from Alfa Aesar, Germany) were used as starting materials. Considering the large particle size of Cr powder (85.5 μm), a planetary milling process (PM400, Retsch, Germany) using Cr-alloy balls (5 mm diameter) for 3 hours at 400 r.p.m. was employed under argon atmosphere to avoid oxygen contamination during the treatment. The final mean particle size was 26.5 μm. Then, the powders were weighed according to the desired composition (Cr:Al:C = 2.0:1.1:1.0) and mixed in a planetary milling for 20 min at 150 r.p.m. under argon atmosphere. An Al excess of 10 at.% was added to compensate its loss during the further thermal treatment. The mixed powder was cold uniaxially pressed at 200 MPa and heat treated under argon atmosphere. The thermal conditions were: heating rate of 5 °C·min⁻¹, maximum temperature between 1100 and 1450 °C, and isothermal holding time of 3 h. Once the pure Cr₂AlC phase was synthesized, the samples were ground in an agate mortar and vibrational milled (Pulverisette Spartan, Fritsch, Germany), obtaining a Cr₂AlC powder with a mean particle size of 9.2 μm.

The Cr₂AlC powder was then mixed with the proper amount of commercial β-SiC fibers (SI-TUFF SF-7, Advanced Composite Materials, LLC, US) to obtain composites containing 5 and 10 wt.% of fibers (hereafter Cr₂AlC/5SiC and Cr₂AlC/10SiC, respectively), which corresponded to a fiber volume content of 8 and 16 %, respectively. The SiC fibers had a mean diameter of 7 μm and length of 70 μm. The Cr₂AlC powder and the SiC fibers were mixed in ethanol combining mechanical stirring and sonication in an ultrasonic bath for 2 h. After the mixing process, the suspension was dried in a rotary evaporator and in an oven at 80 °C overnight. Finally, the dry powder was sieved through 100 μm mesh. Densification of the

composites and the blank specimens (monolithic Cr_2AlC) was carried out using a FAST/SPS (FCT-HPD5, FCT Systeme GmbH, Germany). 4 g of powder were poured into a standard graphite tool of 20 mm inner diameter and pre-compacted under cold uniaxial pressure of 50 MPa during 60 s. Graphite foils were previously placed between the graphite die/punches and the powder to ensure the mechanical, electrical and thermal contact during the sintering. In addition, the graphite system was covered with a porous carbon felt to reduce the heat loss. Temperature was controlled with a pyrometer that was focused on the bottom of the upper punch at a distance of 5 mm from the specimen. Sintering conditions were equal for all the materials: a heating rate of $100\text{ }^\circ\text{C}\cdot\text{min}^{-1}$, maximum temperature of $1250\text{ }^\circ\text{C}$, 10 min of isothermal holding time, and 50 MPa of uniaxial pressure and vacuum ($\sim 3\text{ Pa}$) for the whole sintering process.

Samples were ground to remove the graphite and polished up to $0.5\text{ }\mu\text{m}$ diamond paste. Bulk densities of the 20 mm diameter and $\sim 3\text{ mm}$ thickness disc specimens were measured by the water immersion method at room temperature, and relative densities were calculated by the rule of mixtures using the theoretical values of 5.23 and $3.21\text{ g}\cdot\text{cm}^{-3}$ for Cr_2AlC and SiC fibers, respectively. Phase composition of the starting powders and the sintered specimens was determined by X-ray diffraction (XRD, Advanced Bruker Corporation, Germany). For microstructural analysis, the polished samples were observed using a table top microscope (TM3030, Hitachi, Japan) and a scanning electron microscope (SEM, Zeiss Ultra55, Germany). Grain size was determined over at least 1000 grains from different SEM micrographs by linear intercept method using “lince-Linear Intercept 2.4.2e” software (Nichtmetallisch-anorganische Werkstoffe, FB11, TU Darmstadt, Germany) and a correction factor of 1.56. Matrix/fiber interface and dispersion degree were analyzed by SEM in polished and fracture specimens, whereas the chemical composition of the different phases was identified using energy dispersive spectroscopy (EDS)-connected with SEM.

Mechanical characterization, in particular, Hardness (H) and Elastic modulus (E), was carried out by depth sensing indentation (Zwick/Roell, Zhu 2.5, Germany) using diamond Vickers pyramids and a load of 98 N. E was calculated from the unloading branch of the curve and corrected from the frame compliance of the instrumented indenter. At least six well defined indentations prints were considered per sample. Tribological properties, namely friction coefficient and wear volume, were measured by linear reciprocating ball-on-plate configuration using a UMT-3 tribometer (Bruker Nano Inc., formerly CETR, USA) under dry conditions. Si_3N_4 commercial balls with diameter of 10.319 mm were selected as counterpart. Sliding test conditions were: loads of 2 and 5 N, stroke length of 2.5 mm, frequency of 20

Hz ($0.1 \text{ m}\cdot\text{s}^{-1}$), total sliding distance of 360 m, temperature of $25 \pm 2 \text{ }^{\circ}\text{C}$, and humidity $< 20 \%$. At least three experiments were performed per condition and specimen, and the plotted data correspond with the average values. Friction coefficient (μ) was continuously recorded during the tests, estimating the steady-state value (μ_{ss}) within the interval between 200 and 360 m. After the tests, the linear wear (W_L) was measured in the center of the wear scar using a 2D profilometer (Dektak XT, Bruker, Germany). The wear volume of the plates (W_V) was calculated using the following expression [32]:

$$W_V = \Delta x \times A + \pi \frac{d^2}{8} W_L \quad (1)$$

considering the sum of two terms: the first one represents the main W_V according to the ASTM G133 standard [33], which recommends measuring three 2D profiles for each track to determine the cross section area beneath the 2D curves (A), and then multiply that value by the nominal stroke (Δx). The second term is a correction factor that takes into account the two edges of the wear tracks [34]. This term is assessed considering the width (d) and depth (W_L) of each track. The wear rate of the specimens (W_R) was then calculated as follows:

$$W_R = \frac{W_V}{F_N \cdot s} \quad (2)$$

where F_N the normal force and s the sliding distance. Worn surfaces were characterized by SEM to analyze the role played by the SiC fibers during the tribological response of the $\text{Cr}_2\text{AlC}/\text{SiC}$ composites.

3. Results and discussion

3.1 Processing of $\text{Cr}_2\text{AlC}/\text{SiC}$ composites

The resulting phase composition of Cr_2AlC as a function of the synthesis temperature is shown in Figure 1a. At the lowest temperature, $1100 \text{ }^{\circ}\text{C}$, the Cr_2AlC phase was already formed, although intermetallic and carbide compounds (Cr_2Al and Cr_7C_3 , respectively), and a starting reactant (C) were detected. When the temperature was increased up to $1200 \text{ }^{\circ}\text{C}$, reactions between the intermediate products and the carbon increased the content of Cr_2AlC . At $1300 \text{ }^{\circ}\text{C}$, practically all the reaction intermediates were consumed and only Cr_2AlC and traces of Cr_2Al were detected. Finally, at 1400°C the synthesis reactions finished and pure Cr_2AlC was obtained. However, when the temperature was increased up to $1450 \text{ }^{\circ}\text{C}$ Al_4C_3 phase was identified, suggesting the beginning of the thermal decomposition. The whole reaction sequence during the synthesis process of Cr_2AlC materials is completely described elsewhere by some of the current authors [35]. After the synthesis process, the Cr_2AlC specimen was milled to obtain a fine

1 powder ($d_{50} = 9.2 \mu\text{m}$), retaining the purity of the phase (Fig. 1b). The resulting Cr_2AlC powder exhibited
2 unimodal distribution (not shown in this document) without hard agglomeration (Fig. 2a). In addition,
3 the particles presented a nanolayered structure, as a SEM micrograph at high magnification shows in
4 Fig. 2b. The as-received commercial SiC fibers exhibited no agglomeration too (Fig. 2c), although some
5 SiC and C impurities probably coming from the synthesis and/or processing of the chopped fibers were
6 identified by SEM. Furthermore, the SiC fibers are not fully dense, presenting a porous structure and an
7 unevenness surface (Fig. 2d).

8 Both powders – Cr_2AlC and SiC fibers – were successfully mixed in a proper amount to obtain the desired
9 $\text{Cr}_2\text{AlC}/5\text{SiC}$ and $\text{Cr}_2\text{AlC}/10\text{SiC}$ powder compositions, and then, sintered by FAST/SPS. Cr_2AlC and SiC
10 were the only phases detected by XRD in the densified samples (Fig. 1b), confirming the absence of
11 secondary phase formation by reaction or degradation. Table I shows the density, relative density and
12 grain size of all the sintered materials, as well as hardness and elastic modulus. All the samples exhibited
13 high density ($\geq 98.5\%$ of the theoretical value, ρ_{th}), the monolithic material ($\rho_{\text{th}} = 98.9$) being slightly
14 denser than the composites because the presence of the elongated and stiff SiC fibers in the latter
15 materials partially hinder the densification. Pure Cr_2AlC material presented the highest d_{50} value (15.1
16 μm), whereas $\text{Cr}_2\text{AlC}/5\text{SiC}$ and $\text{Cr}_2\text{AlC}/10\text{SiC}$ exhibited smaller grain sizes, in particular, 13.8 and 13.6
17 μm , respectively. As for the densification process, the lower d_{50} values for the composites were related
18 with the hindering of the grain growth process by the SiC fibers during the last step of the densification
19 process. Nevertheless, small differences in density and grain size were obtained.

20 One of the critical aspects for developing the composite materials with different particle shapes is the
21 dispersion of the second phase within the matrix. In that sense, the degree of dispersion of the
22 composites was analyzed through the observation of polished and fracture surfaces (Figure 3). Pale gray
23 areas correspond with the Cr_2AlC matrix, whereas the dark gray features are the SiC fibers. The
24 composites presented an excellent dispersion of the SiC fibers within the matrix (Figs. 3a-c), which were
25 perpendicularly oriented to the FAST/SPS pressing axis, leading to an anisotropic composite. This effect
26 is commonly observed during the densification by FAST/SPS – or other uniaxially pressed techniques, i.e.
27 HP - of composites with elongated second phases [36]. Furthermore, the fibers showed two different
28 regions associated to distinct materials, a black core of carbon and an outer dark gray ring of SiC. The
29 fracture of $\text{Cr}_2\text{AlC}/10\text{SiC}$ (Fig. 3c) exhibits a relative smooth surface and the absence of fiber pull-out,
30 which can be explained by a strong bonding interface between the matrix and the fiber. In fact, SEM
31 observations at higher magnification of the fracture surface for both composites (Figs. 3d and e)

confirmed the strong matrix/fiber bonding, causing the fiber breakage. Furthermore, the matrix presented transgranular fracture mode, where the nanolayered structure of the Cr_2AlC grains was clearly observed (Fig. 3f), some of them being also kinked and delaminated.

Matrix/fiber interface and reactivity between them were analyzed by EDS on polished specimens (Fig. 4). Interestingly, no reaction was observed between the matrix and fibers, avoiding the formation of an interface reactive layer. This reactive layer was observed by Guo et al.[29] in HPed $\text{Ti}_3\text{SiC}_2/\text{SiC}$ composites and was consisted of brittle intermetallic compounds that reduced the mechanical properties. The solution to avoid this reaction was to use SiC fibers with an outermost coating layer richer in carbon than the stoichiometric SiC fiber [29], the so called SCS layer. In the present case, the diffusion behavior of alloying elements was suppressed by using FAST/SPS as sintering technique instead HP, limiting the time at the maximum temperature to minutes instead hours. The SiC fibers were entirely surrounded by the Cr_2AlC matrix. In addition, neither cracks nor pores were observed in the vicinity of the fibers and no interface reactive layer between the matrix/fiber was detected. The core of the fibers exhibited a strong signal linked to carbon element (region I). It should be noted that carbon is difficult to detect by EDS due to its low atomic number, but its high concentration in region I facilitates its detection. Fibers were mostly formed by SiC (region II), although small contents of chromium and aluminum were also detected. These elements can diffuse from the Cr_2AlC to the porous fibers. Two different areas were observed in the interface between the matrix and the fiber (regions III and IV). Region III presented a high content of silicon and carbon, which could be corresponded to SiC particles (in dark) presented in the commercial SiC fibers, as observed in Fig. 2c. The lighter gray particle (region IV) contained large amount of chromium, aluminum, carbon and silicon. Thus, this particle was non-stoichiometric Cr-Al-Si-C compound, where the silicon could be incorporated to the Cr_2AlC -MAX cell on Al position. Finally, the matrix (region V) exhibited the characteristic Cr_2AlC phase. As a result, we can conclude that no reactions between the matrix and the fibers occurred during the sintering process, although low diffusion of aluminum and chromium atoms was detected into the fibers.

3.2. Tribological properties

Figure 5 shows the friction coefficient evolution as a function of the sliding distance and applied load (Figs. 5a and b), as well as the steady-state friction coefficient versus the SiC fibers content (Fig. 5c). At 2 N, the benefits of adding SiC fibers are clearly observed from the very beginning of the sliding tests (Fig. 5a), decreasing the friction coefficient at the first instants (< 20 m) from 1.6, for the monolithic material, to 1.3, for the $\text{Cr}_2\text{AlC}/10\text{SiC}$ composite. As the sliding test progressed, this effect was maintained and

the μ_{ss} value of SiC diminished about 20% (from 2 to 1.6) with the incorporation of 10 wt.% of SiC fibers (Fig. 5c). Cr₂AlC monolithic material showed unevenness μ during the whole sliding test, as it was observed between 15-40 m and stronger between 220-280 m. This effect was already reported by Gupta et al. [24] and associated with the discontinuous and non-uniform nature of the tribofilm because of the low oxidation kinetics of Cr₂AlC debris. When the load increased up to 5 N, the μ_{ss} for all the materials was reduced as compared to 2 N values (Fig. 5c), especially for the monolithic material, which also presented more stable and smoother μ . Besides, the influence of the SiC fibers in the friction response vanished, as μ_{ss} remained invariable with the SiC content ($\mu_{ss} \sim 1.5$).

At the first glance, from the 2D profiles at the middle of the wear tracks at 2 and 5 N of all the tested materials (Fig. 6), it seems that the wear behavior of the Cr₂AlC materials was undeniably improved by the incorporation of SiC fibers. To quantify that improvement, W_v and W_R values were calculated (Fig. 7). In this way, monolithic Cr₂AlC was eroded 0.13 mm³ under 2 N, whereas 0.050 and 0.025 mm³ were removed for Cr₂AlC/5SiC and Cr₂AlC/10SiC, respectively (Fig. 7a), which represents a decrease on W_v of 57 and 80 %. The results when using a load of 5 N show an overall increase of the W_v for all the materials, although the addition of SiC fibers again led to a better wear response, in particular, up to 67% for the composite containing 10 wt.% of SiC fibers as compared to the monolithic material (Fig. 7a). The wear rate behavior follows a similar trend with the applied load and the fibers content than that for the wear volume, with W_R values in the order of 10⁻⁴-10⁻⁵ mm³·N⁻¹·m⁻¹ (Fig. 7b) and a maximum reduction of 80% attained for the Cr₂AlC/10SiC composite.

The worn surfaces of the tested materials at the different loads were observed by SEM (Fig. 8) and a severe wear process was clearly identified for all of them, which is in good agreement with the W_R exponents. At 2 N, the wear track of the monolithic material exhibited an extensive cracked debris layer coming from the pulling-out and crashing of the Cr₂AlC grains (Fig. 8a); whereas the wear track of the composites also showed those areas covered by debris as well as smoother regions where the polished surfaces presented some abrasive grooves (Fig. 8c) but with signs of fracture. These grooved surfaces enlarged with the SiC fiber contents, limiting the wear volume (Fig. 7) and reducing the friction (Fig. 5c) as compared to the worn Cr₂AlC surface. At higher loads (5 N), the debris layer on the monolithic Cr₂AlC became unstable and was mostly removed, exposing the fractured surface of the material (Fig. 8d). However, the debris layer was in the forming stage for the Cr₂AlC/10SiC composite (Fig. 8f), similarly to the situation exhibited for the monolithic material at the lowest load and showing quite close W_v values (0.13 mm³ for Cr₂AlC at 2 N versus 0.15 mm³ for Cr₂AlC/10SiC at 5 N). The Cr₂AlC/5SiC composite

exhibited an intermediate situation with fractured regions and stable debris layer areas (Fig. 8e). Therefore, it can be concluded that abrasion is the main wear mechanisms although surface fatigue also occurred. One possible explanation for the enhanced wear resistance of the SiC fiber containing composites could be associated to their better mechanical performance. Actually, the abrasive wear resistance is commonly related to the hardness and fracture toughness, W_v being proportional to $H^{-1/2} \times K_{IC}^{-3/4}$ [37]. Therefore, the higher hardness exhibited by the Cr_2AlC/SiC composites (Table 1) in conjunction with the expected toughening provided by the SiC fibers [31] would substantially reduce the wear volume.

Conclusions

Homogeneous and highly dense Cr_2AlC composites containing 5 and 10 wt.% of SiC fibers have been successfully processed for the first time. The processing route is simple and cheap, and consists in two consecutive steps: i) synthesis of the pure Cr_2AlC phase at 1400 °C under argon atmosphere and processing of the Cr_2AlC/SiC fibers powders composition, and ii) FAST/SPS densification. No reaction between the Cr_2AlC and the SiC fibers takes place due to the FAST/SPS sintering, although a strong bonding matrix/fiber was observed, causing the fiber breakage instead reinforced mechanisms. The incorporation of the SiC fibers enhances the tribological behavior of the Cr_2AlC materials under dry conditions, in particular, the friction decreases up to 20% while the wear resistance increases up to 80% for $Cr_2AlC/10SiC$ composites. This response is linked to the better mechanical performance of the composites as compared to the monolithic Cr_2AlC .

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Figure and table caption

Figure 1. XRD patterns of a) synthesis of Cr₂AlC at 1100 – 1450 °C for 3 hours, and b) synthesized Cr₂AlC at 1400 °C, the milled Cr₂AlC powder and the densified Cr₂AlC/SiC composite by FAST/SPS.

Figure 2. SEM micrographs of a) and b) synthesized and milled Cr₂AlC powder, and c) and d) commercial SiC fibers.

Figure 3. SEM micrographs of polished a) top surface, and b) cross section of Cr₂AlC/10SiC, and fracture surface of c), e) and f) Cr₂AlC/10SiC and d) Cr₂AlC/5SiC.

Figure 4. SEM micrograph of polished cross section of Cr₂AlC/10SiC and EDS on regions marked (I-V).

Figure 5. Friction coefficient evolution for the different materials under load of a) 2N and b) 5N, and c) friction coefficient as a function of the fiber content for both loads.

Figure 6. 2D profiles of the wear tracks of the different materials at a) 2N and b) 5N.

Figure 7. a) Wear volume and b) wear rate for all the tested materials at 2N and 5N.

Figure 8. SEM micrographs of the worn surfaces tested at 2 N (a-c) and 5 N (d-f) for the following materials: a,d) monolithic Cr₂AlC, b,e) Cr₂AlC/5SiC composite, and d,f) Cr₂AlC/10SiC composite.

Table I. Density, relative density, mean grain size, hardness (H) and elastic modulus (E) of the sintered materials.

Sample	Density (g/cm ³)	Relative density (%)	Grain size (μm)	H (GPa)	E (GPa)
Cr ₂ AlC	5.17	98.9	15.1 ± 0.2	4.12 ± 0.27	262 ± 19
Cr ₂ AlC/5SiC	5.06	98.6	13.8 ± 0.1	5.08 ± 0.30	235 ± 38
Cr ₂ AlC/10SiC	4.95	98.5	13.6 ± 0.1	5.85 ± 0.29	224 ± 50

Figure 1a

Figure 1b

Figure 2
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Figure 3
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Figure 4
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Figure 5a

Figure 5b

Figure 5c

Figure 6a

Figure 6b

Figure 7a

Figure 7b

Figure 8
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