MAX phases as candidates for Concentrated Solar Power receivers

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Abstract

New generation concentrated solar power (CSP) plants require new solar receiver materials with selective optical properties and excellent corrosion resistance against molten salts. MAX phases are promising materials for CSP applications due to their optical properties and resistance to extreme environments. Herein, we report a solar absorptance \( \geq 0.5 \) and a thermal emittance of 0.17-0.31 between 600 and 1500 K for \( \text{Cr}_2\text{AlC} \), \( \text{Ti}_2\text{AlC} \), and \( \text{Ti}_3\text{AlC}_2 \). These compositions were also exposed to solar salt corrosion at 600 °C for up to 4 weeks. \( \text{Cr}_2\text{AlC} \) exhibited a superior corrosion resistance due to the formation of a protective nanometric layer.

Keywords:

MAX phases; concentrated solar power; corrosion; solar receivers; optical properties.
Impact Statement
We show that MAX phases are promising replacement materials for current solar receivers/absorbers in view of their optical performance and better corrosion resistance to solar salt.

1. Introduction
Concentrated solar power (CSP) is attracting major attention as we are progressing towards more sustainable, safer, and cost-effective technologies for electricity production. The efficiency of CSP systems is rapidly increasing with increasing operation temperature, requiring hence, materials with good thermal stability, oxidation resistance, and preserved spectral selectivity, to name a few. In that regard, two different CSP receiver concepts have been developed to increase the operating temperature, where the difference resides in the Heat Transfer Fluid (HTF). The first CSP concept uses molten salt as HTF, so the corrosion resistance of the solar receivers and heat exchangers is typically the main limitation. The second CSP approach uses volumetric air receivers, where air is the HTF and the main challenge in terms of material properties lies in the optical response and oxidation resistance. Recently, ultra-high temperature ceramics (UHTCs), and in particular diborides [1–4] and carbides [3,5] have been suggested as suitable alternatives to the more conventional graphite [6] absorbers. Other promising materials which fulfill the specification requirements and which have yet to be fully investigated, are MAX phases.

MAX phases are inherently nanolaminated ternary carbides and/or nitrides with the $M_{n+1}AX_n$ ($n=1,2,3$) general formula, where $M$ is an early transition metal, $A$ is an A group element, and $X$ is carbon or nitrogen [7–9]. The chemical versatility and structure of these MAX phases provide the materials with a unique combination of metallic and ceramic
properties, making them interesting for a variety of applications [10–12]. MAX phases have shown great promise for high temperature applications, because of their excellent mechanical performance in oxidizing and corrosive environments [13–18], as well as their self-healing and self-protective ability [13,19,20]. In terms of mechanical response, Barua et al. recently showed that Ti-based MAX phases would extend the operating time of the receivers for Gen3 CSP towers as their creep resistance at high temperature surpasses that of A740H nickel-based alloy structural materials [17]. Regarding the thermal performance and optical properties, Sarwar et al. carried out studies on Cr$_2$AlC and Ti$_2$AlC under high flux solar simulator and assessed the promising nature of both MAX phases [21]. However, the most critical factor for CSP materials is the hot corrosion resistance against molten salts. Van Loo et al. investigated the interaction of different SiC grades, metallic alloys, and several MAX phases with a KNO$_3$-NaNO$_3$ solar salt at 600 °C. Cr$_2$AlC exhibited the best performance of all tested compositions due to the formation of a stable and sub-micrometer-thick Cr$_7$C$_3$ scale, which strongly minimized the interaction with molten salts [22]. Additional studies are certainly needed, but these preliminary works have shown high potential for MAX phases to increase the efficiency and operating lifetime of CSP systems.

Herein, we studied three MAX phases, namely Ti$_2$AlC, Ti$_3$AlC$_2$, and Cr$_2$AlC, as candidates for solar absorbers in CSP technologies with preliminary promising outcomes [21,22]. The optical properties of the as-prepared MAX phases were investigated to compare their performance with that of selected diborides and SiC, while the corrosion resistance of these MAX phases in solar salt at 600 °C was assessed for up to 672 h.
2. Materials and Methods

Ti$_2$AlC, Ti$_3$AlC$_2$, and Cr$_2$AlC fully dense samples were prepared in a two-step process. First, powders were prepared either by the molten salt shielded synthesis process (Ti$_2$AlC) [23], or by solid-state reaction at high temperatures (Ti$_3$AlC$_2$, Cr$_2$AlC) [24]. Afterward, the powders were sintered into 30 mm diameter discs by Field Assisted Sintering Technology/Spark Plasma Sintering (FAST/SPS) using standard graphite tools and the same set of parameters of 100 K/min heating rate, 1200 °C maximum temperature held for 10 min, vacuum < 3Pa, and 50 MPa uniaxial pressure applied during the whole thermal treatment. After densification, the samples were ground to remove the protective graphite foil, followed by fine grinding to 6 μm with P4000 SiC paper, leaving a final sample thickness of ca. 4 mm.

The average surface roughness of each sample was determined in the center of each disc using a Contour GT-K 3D non-contact profilometer (Bruker, Germany) on areas of 6×6 mm$^2$ and the data were analyzed using commercial software (Vision64 Map). The evaluation of 2D texture parameters (average roughness, $R_a$, and maximum distance between peak and valley, $R_t$) was performed on 4 different profiles extracted from the 3D data, and the gaussian filter ($k_c$) for the separation of the roughness and waviness components were set according to the ISO 4287.

Optical reflectance spectra at room temperature and quasi-normal incidence angle in the 0.25-2.5 μm wavelength region were acquired using a double-beam spectrophotometer (Perkin Elmer Lambda900) equipped with a 150 mm in diameter integration sphere for the measurement of the hemispherical reflectance. The spectra in the wavelength region 2.5-15.5 μm were acquired using a Fourier Transform
spectrophotometer (FT-IR Bio-Rad Excalibur) equipped with a gold-coated integrating sphere and a liquid nitrogen-cooled detector.

The corrosion tests were performed, on ca. 6.5×6.5×4.1 mm³ blocks, as follows: initially, sodium nitrate (NaNO₃, 98+%, Alfa Aesar) and potassium nitrate (KNO₃, 99%, Alfa Aesar) were mixed in a 60:40 weight ratio, corresponding to the eutectic molten salt mixture (referenced as “solar salt”). The MAX phase samples were placed in alumina crucibles and covered with the blend. The blocks were corroded for up to 4 weeks, at 600 °C (10 K/min). The samples were removed from the solar salt after 24, 168, 336, and 672 hours, and further characterized.

Following the approach adopted in corrosion tests of Al₂O₃-forming steel [25] and Ti-based MAX phases [14,26–28], the Ti-based samples were pre-oxidized in air at 1100 °C for 12h (10 K/min), to form dense protective Al₂O₃ scales of ~2.2 and ~1.9 μm in thickness, respectively, as estimated by cross-section thickness measurements, and thus prevent the formation of non-protective TiO₂, Na₂(TiO₃), and Na₂(TiSi₅O₁₃), products reported by Van Loo et al. [22].

Scanning electron microscopy (SEM, Zeiss Crossbeam 540, Carl Zeiss AG, Germany) was carried out on embedded and polished samples for the microstructural characterization before and after the corrosion tests. Scanning transmission electron microscopy (STEM) was carried out on thin lamellae that were prepared in a FEI Helios NanoLab dual-beam focused ion beam (FIB) microscope, using Ga⁺ ions accelerated at 30 kV. A STEM III detector was used for imaging. Line scans were collected using an EDAX Octane Elect EDX detector, using an accelerating voltage of 12 kV.
3. Results and discussion

Table 1 gathers the densities, phases identified by X-ray diffraction and refined by Rietveld analysis, and the average surface roughness, $R_a$ of the as-sintered MAX phases. Sintering led to fully dense MAX phase bulks, achieving relative density values above 99%. These high values are confirmed in the SEM pictures of the polished surfaces (Fig. 1), where almost no porosity is observed. The light grey phases detected in the back-scattered SEM micrographs correspond to the MAX phase, while the darker grey phases are Al$_2$O$_3$ particles. The content of these phases was evaluated by Rietveld analysis and included in Table 1. Al$_2$O$_3$ was detected in all the MAX phase compositions, which was correlated to the inherent surface oxidation of the starting Al powder used as reactant. Small amounts of Al$_2$O$_3$ and Ti$_2$AlC were detected in Ti$_3$AlC$_2$ (Fig. 1(b)), but also trace amounts of Fe-based compounds found adjacent to the oxide phase. These Fe-compounds could not be correlated to impurities of the starting powders as they are not detected in the Ti$_2$AlC samples, so we assume they were incorporated during the milling process. Cr$_2$AlC samples exhibit high purity, presenting just 4% of Al$_2$O$_3$ and 1% of Cr$_7$C$_3$ (Fig. 1(c)).

Regarding the average surface roughness, while it is in the same order of magnitude for the three MAX phases, Cr$_2$AlC appears to have the lowest roughness, possibly because of the presence of less secondary phases and therefore less pull out caused by polishing.

Table 1: Experimental relative density, phase composition and grain size of the MAX phases sintered by SPS at 1200°C, for 10 min dwell under 50 MPa applied pressure, and average roughness (Ra) of the polished samples.

| Material | Relative density (%) | Crystalline Phases by Rietveld analysis (wt.%) | $R_a$ (nm) |
|----------|----------------------|-----------------------------------------------|------------|
|          |                      |                                               |            |


Once the MAX phase samples were mirror polished, hemispherical reflectance spectra were measured in the full spectral range 0.3-16.0 μm (Fig. 2(a)) and in the solar spectrum region, 0.3-3.0 μm wavelength (Fig. 2(b)). All three MAX phases show step-like spectra indicating intrinsic spectral selectivity. Ti$_2$AlC and Ti$_3$AlC$_2$ spectra nearly superimpose each other in the infrared from 3 μm on, while some minor spectral differences can be seen at shorter wavelengths (Fig. 2(b)). Cr$_2$AlC has a slightly lower reflectance up to about 10 μm and a curve rise front slightly blueshifted with respect to the Ti-based samples.

From hemispherical reflectance spectra $\rho(\lambda)$, it is possible to estimate the total hemispherical emittance, $\varepsilon$, at temperature $T$ and the total solar absorptance, $\alpha$, according to the following equations:

$$
\varepsilon = \frac{\int_a^b (1 - \rho(\lambda)) \cdot B(\lambda, T) d\lambda}{\int_a^b B(\lambda, T) d\lambda} 
$$

$$
\alpha = \frac{\int_a^b \rho(\lambda) \cdot B(\lambda, T) d\lambda}{\int_a^b B(\lambda, T) d\lambda} 
$$

(1)
where \( B(\lambda, T) \) is the blackbody spectral radiance at the temperature \( T \) of interest and the integration bounds are \( \lambda_1 = 0.3 \) µm and \( \lambda_2 = 16.0 \) µm.

Similarly, the total solar absorptance can be calculated as:

\[
\alpha = \frac{\int_{\lambda_{\text{min}}}^{\lambda_{\text{max}}} (1 - \rho^\alpha(\lambda)) \cdot S(\lambda) \, d\lambda}{\int_{\lambda_{\text{min}}}^{\lambda_{\text{max}}} S(\lambda) \, d\lambda}
\]

where \( S(\lambda) \) is the A.M. 1.5 sunlight spectral distribution [29] and the integration is carried out between \( \lambda_{\text{min}}=0.3 \) µm and \( \lambda_{\text{max}}=3.0 \) µm.

The two main optical parameters to consider for solar applications are the solar absorptance, \( \alpha \), and the \( \alpha/\varepsilon \) ratio or spectral selectivity. In an ideal spectrally-selective material, \( \alpha \) should be the nearest as possible to unity, while \( \alpha/\varepsilon \) should be taken as high as possible. Non-selective (so-called grey) materials, like silicon carbide (SiC), show \( \alpha/\varepsilon \) values near to unity. In the case of MAX phases, the \( \alpha/\varepsilon \) values are not reported in the literature to the best of the authors’ knowledge.

Fig. 2(c) shows the temperature-dependent emittance values calculated for the MAX phase samples compared to those of a fully dense SiC reference and to other transition metal borides [4,30]. The values of solar absorptance are also indicated in the figure caption, while the temperature-dependent spectral selectivity values are plotted in Fig. 2(d). The three MAX phases show similar properties, with \( \alpha \) values \( \geq 0.5 \) and \( \varepsilon \) in the 0.17-0.31 range at the considered temperatures. The sample with the highest solar absorptance and emittance is \( \text{Cr}_2\text{AlC} \), corresponding to a slightly lower spectral selectivity.
than Ti-containing compounds for temperatures below 1100 K. At higher temperatures, the three samples are equivalent.

As for the comparison with previously investigated borides and SiC, the latter being currently the most advanced absorber used in solar plants, MAX phases are significantly more spectrally selective than SiC, and, like borides, have a lower solar absorptance. However, as we previously reported, this weakness can be mitigated with a selective increase of solar absorptance through proper surface texturing techniques [31,32].

Figure 2: (a) Hemispherical reflectance spectra of MAX samples in the 0.3-16 μm range and (b) detail of the solar spectrum range. (c) Calculated total emittance and (d) calculated spectral selectivity in the 600-1500 K temperature range. Data for SiC, HfB₂ and ZrB₂ reference materials are taken from [30] and [4]. For a fair comparison, since surface finishing and possible porosity have an impact on the resulting optical properties, the materials here considered for reference are all dense and polished.
As discussed above, the optical properties of the different MAX phases show high potential for using these compositions in CSP applications, in particular for volumetric air receivers. Likewise, another relevant factor – mainly for molten salt CSP systems – is corrosion resistance at high temperature. Accordingly, the three MAX phases were corroded by solar salt at 600 °C for up to 4 weeks (672 h), followed by microstructural characterization. The XRD pattern of the as-sintered Ti$_2$AlC sample are provided in Fig. 3(a), as well as those upon corrosion for 168, 336 and 672 h. After 168 h corrosion, peaks corresponding to Al$_2$O$_3$, Na$_5$AlO$_4$, NaTi$_2$O$_4$, and NaAl$_6$O$_{9.5}$ were detected. The intensity of those peaks increased with increasing corrosion time, indicating that larger amounts of corrosion products formed. Furthermore, one can notice that the MAX phase peaks disappeared almost completely after 168 h, hinting at a considerable thickness of corrosion layer, which was then confirmed by cross-section imaging, as shown in Fig. 3(b)-(d). Indeed, the corrosion layer was nearly 10 μm-thick even after 24 h, and inhomogeneous as some parts were less than 1 μm-thick. The scale became more homogeneous in thickness after 336 h and remained stable at ~10 μm. The scale appeared to be somewhat lamellar and quite porous, suggesting it might perform poorly in terms of corrosion protection. As the corrosion time reached 672 h, the thickness of the scale averaged at 20 μm. Chemical mapping (EDS) performed over the area shown in Fig. 3(e), showed that the scale contains primarily Ti, Na and Ca, whereas Al was detected on the outmost surface scale.

Overall, these observations led to conclude that the Al$_2$O$_3$ scale grown prior to the corrosion tests did not yield the expected protective effect. In fact, the alumina layer may have behaved in two different ways. First, the oxide scale could have been consumed by the solar salt. This would support the reports from Van Loo et al. who did not identify Al$_2$O$_3$
after corrosion of untreated (not pre-oxidized) commercial Maxthal® Ti₂AlC and Ti₃AlC₂ samples in the same solar salt for up to 1000 h [22]. The second scenario would imply spalling of the alumina scale, which is supported by Al-rich leftovers seen in Fig. 3(e). Considering that the thickness of the Al₂O₃ layer was rather thin, close to 2 μm, the stresses generated during corrosion may have led to the premature fragmentation of the scale [33]. The salt could have, then, easily corroded the underlying MAX phase. This hypothesis seems to be more reasonable as it is supported by microstructural findings in Fig. 3. Finally, despite the presence of significant amounts of corrosion products, the Ti₂AlC sample exhibited a good resistance to the solar salt as it maintained its integrity even after 672 h, as illustrated in the inset of Fig. 3(a).

Figure 3: (a) XRD patterns of as-sintered and corroded Ti₂AlC samples. (b)-(d) cross-section micrographs of corroded samples after 24, 336 and 672 h. (e) Magnified image and EDS elemental...
maps of the rectangular zone in (d). The picture in (a) shows the appearance of the corroded 6.5×6.5×4.1 cm³ sample after 672 h corrosion.

Similar results might be expected for Ti₃AlC₂ due to the same elements, however the resistance towards the solar salt differed significantly. As it can be seen from the XRD patterns in Fig. 4(a), a multitude of peaks corresponding to various oxides appeared after 168 h. In this case, aside from Na-containing aluminides and titanates, TiO₂ was indexed, unlike the case of Ti₂AlC, which may be expect to nucleate with more ease when from Ti₃AlC₂ because of the lower Al content. The poorer corrosion resistance of Ti₃AlC₂ was further demonstrated by the degradation of the sample integrity with corrosion time (see Fig. 4(b)). The corrosion initiated at the corners and edges, where thermal and mechanical stresses between the pristine sample and corrosion layer are greater. After 672h, the sample completely disintegrated. The cross-section micrograph of the sample corroded for 24 h is shown in Figure 4(c). While the corrosion scale seemed relatively dense, its thickness had already reached ~68 μm, confirming the poor corrosion resistance of Ti₃AlC₂ in solar salt. From the EDS maps, one can notice the absence of the continuous Al₂O₃ scale formed prior to the corrosion tests, but rather the presence of Al-rich grains which could be leftovers of the intentionally formed Al₂O₃ scale. Furthermore, one can notice Ti-rich zones in the scale indicating that the composition within the scale is inhomogeneous. In fact, a chemical composition contrast can also be seen on the micrograph in Figure 2(c). In this case, K was also detected in the reaction layer, which was not detected in the reaction layer of the Ti₂AlC MAX phase.
Such behavior was not observed in the case of Cr$_2$AlC. This MAX phase does not appear to have interacted with the solar salt even after 672 h, as confirmed by both the XRD patterns and the SEM cross-section micrographs in Fig. 5(a)-(d), indicating excellent resistance to solar salt. In fact, the formed scale was ~245 nm-thick after 168 h, as measured from STEM imaging (Fig. 5(d)). Van Loo et al. reported similar behavior between Cr$_2$AlC and the solar salt [22]. The formation of a dense scale filling even the crevasses of the MAX phase can be seen in Fig. 5(e). The EDS line scan in Fig. 5(f) shows that the oxide scale is fairly complex. Cr, Na and C enrichment can be noticed in the outermost surface; however, the thin scale was primarily Al-based. This seems to be in contrast with the observations made in the case of the Ti-based MAX phases, as Al$_2$O$_3$
was not identified. However, the Cr-rich outermost surface may have also played an important role in the corrosion resistance of the sample. The corrosion resistance of a stainless Cr-containing steel, AISI 304 L, was shown to be superior than that of a Cr-free carbon steel, ASTM A36, as the corrosion scales after 21 days reached 158 and 610 nm, respectively in solar salt at 390 °C [34]. Compositional depth profiles of the steel samples showed that Cr diffused to the surface and its content increased with increasing corrosion time. In contrast, it has also been reported [35,36] that Cr is actually dissolved by the solar salt, indicating that its role in the protection of steels is almost null. However, it is difficult at this stage to make any conclusive statement as the chemical environment of Cr in stainless steel differs significantly from that in Cr2AlC. Based on the observations made by Van Loo et al., the possibility of forming a Cr7C3 outer layer, with no significant oxygen uptake, would explain the enhanced corrosion resistance of Cr2AlC [22]. However, in our case, a significant amount of oxygen was detected in the outermost thin scale (see Fig. 5(e), (f)) suggesting the presence of an oxide phase. Furthermore, Mg, Na and Si were also detected in the corrosion layer. Further focused investigation is ongoing to reveal the nature of this thin protective layer, which showed an excellent corrosion resistance towards the solar salt based on the only reported publication in the literature [22] and the current work.
Figure 5: (a) XRD patterns of Cr$_2$AlC samples before and after 168, 332, and 672h corrosion in solar salt. SEM cross-section micrographs of Cr$_2$AlC samples corroded for (b) 24, (c) 336 and (d) 672h in solar salt. (e) HAADF STEM image of Cr$_2$AlC corroded for 168 h in solar salt, (f) EDS line scan collected along the dashed line in (e), (g) high magnification micrograph of the corrosion layer in HAADF mode corresponding to the dashed frame in (e).

4. Conclusion

The optical properties and corrosion resistance against the solar salt at high temperature of Ti$_2$AlC, Ti$_3$AlC$_2$ and Cr$_2$AlC MAX phases were investigated to evaluate their potential for use in concentrated solar power receivers. The three materials presented similar optical responses, with solar absorptance $\geq 0.5$ and thermal emittance in the 0.17-0.31 range between 600 and 1500 K. These solar absorptance values were slightly higher than those of ZrB$_2$ and HfB$_2$ (0.48 and 0.47, respectively), but lower than SiC (0.79). Likewise, the thermal emittance of MAX phases lied between SiC (~0.8) and ZrB$_2$ and HfB$_2$ (0.06-0.23). The spectral selectivity values ($\alpha/\varepsilon$) of MAX phases were quite similar and ranged between those of SiC and the borides. Therefore, we can conclude that the three MAX phase compositions present relevant optical properties for further development in CSP systems, in particular for volumetric air receivers where the optical properties are more determinant.
The corrosion resistance of the three MAX phases was also tested in a typical solar salt NaNO$_3$-KNO$_3$ mixture at 600 °C for up to 4 weeks. Ti$_2$AlC and Cr$_2$AlC performed quite well, while Ti$_3$AlC$_2$ fully decomposed. In particular, Cr$_2$AlC exhibited minimal signs of corrosion after 4 weeks. A thin corrosion layer of ~245 nm formed after 168 h, while that of Ti$_2$AlC was already close to 10 μm-thick after just 24 h. While the corrosion layer thicknesses differed, both Ti$_2$AlC and Cr$_2$AlC MAX phases exhibited good corrosion resistance in solar salt making them relevant for application as molten-salt receivers and heat exchangers.

**Acknowledgments**

C.A. acknowledges funding from the European Union’s H2020-MSCA-IF-2019 research and innovation programme under the Marie Skłodowska-Curie grant agreement No. 892501 (REALMAX). The authors would like to thank Ms Melina Poll for sintering the samples and Prof. Jochen M. Schneider for access to the Focused Ion Beam (FIB).

**Declaration of interest statement**

The authors confirm that there are no known conflicts of interest associated with this publication.

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