On the Interactions of Ti_2AlC , Ti_3AlC_2 , Ti_3SiC_2 and Cr_2AlC with Pure Sodium at 550 °C and 750 °C

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Abstract

Herein we report on the interaction of the $M_{n+1}AX_n$ phases – Ti_2AlC , Ti_3AlC_2 , Ti_3SiC_2 and Cr_2AlC – with static, pure molten sodium. The MAX phases were sealed in 316 stainless steel tubes filled with Na that, in turn, were heated to 550 °C or 750 °C for 168 h. Based on scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy dispersive X-ray spectroscopy (EDX), and X-ray diffraction (XRD) of post-exposed samples we conclude that even after exposure for 168 h at 750 °C, Ti_2AlC , Ti_3SiC_2 and Cr_2AlC did not react with Na. In the case of Ti_3AlC_2 , exposure to molten Na alters both the grains and the grain boundaries, despite the fact that no Na signal was detected in the post-exposed samples. These chemical alterations were observed in TEM and manifested themselves by a change in the hardness values of the exposed surfaces.

I. Introduction

The $M_{n+1}AX_n$ phases are ternary, layered machinable carbides and nitrides, where M is an early transition metal, A is a group A element, mostly from groups 13 and 14, and X is either C and/or N [1]. They possess a hexagonal crystal structure, and belong to the $P6_3/mmc$ space group [1]. The latter can be further categorized by their n value as “211s” for M_2AX ($n = 1$), “312s” for M_3AX_2 ($n = 2$) and “413s” for M_4AX_3 ($n = 3$), etc. [2]. To date over 70 known MAX phases are known to exist. These materials combine some of the more attractive properties of ceramics and metals. As with most metals, they have high electric and thermal conductivities[3-5], are easily machinable [6], and possess exceptional thermal shock resistance and damage tolerance[3,5,7]. Some MAX phases, such as Ti_2AlC and Ti_3SiC_2 , are lightweight, stiff, creep [8-12], oxidation [13-17], and fatigue [18] resistant.

When heated in air, Ti_2AlC and Ti_3AlC_2 form thin alumina protective layers and thus have excellent oxidation resistances at temperature up to 1400 °C, even under severe cyclic heating conditions [16]. The same compounds are also stable in vacuum to temperature up to 1400 °C [17]. In the case of Cr_2AlC , at 1000 and 1100 °C, the oxidation resistance is also quite good, because a thin alumina layer forms. In this case a narrow Cr_7C_3 underlayer also forms, below the alumina protective oxide [19]. At 1200 °C and 1300 °C, an outer $(Al_{1-x},Cr_x)_2O_3$ mixed oxide layer, an intermediate Cr_2O_3 oxide layer, an inner Al_2O_3 oxide layer, and a Cr_7C_3 underlayer form. At this temperature, spallation occurs, limiting the use of this material to temperatures no higher than 1100 °C, or even 1000 °C [20].

Currently, there is little research in literature on the chemical stability of the MAX phases vis-à-vis materials currently used or proposed for use in nuclear energy systems. For example, before the MAX phases can be considered for use in Gen IV

nuclear reactors and to better understand where they would be most judiciously used, much needs to be better understood and quantified. A systematic research effort into their chemical stability/reactivity with key materials used or proposed in nuclear energy production, such as high temperature structural components or diffusion barrier coatings for nuclear fuels, is therefore required.

Coolants that have attracted interest for the next generation nuclear reactors includes, He gas, molten Na, Pb-Bi and FLiBe [21-23]. Certain MAX phases have been tested for corrosion in select coolant environments. For example, it has been shown that Ti_3SiC_2 has superb corrosion resistance vis-à-vis molten Pb and Pb-Bi alloys [24,25]. Over the last 50 years, the idea of liquid metal cooled fast reactors (LMFR) has been conceived, researched, and even implemented [26]. Due to its abundance and relative low cost compared to other coolants, liquid Na has been considered as an attractive coolant [27]. Over the years, there have been experimental Na cooled LMFRs in France (Rapsodie), Germany (KNK-II), India (FBTR), Japan (JOYO), Russia (BOR-60 and BR-10), the US (EBR-II, Fermi, and FFTF), and one currently under construction in China (CEFR). Of the commercial liquid cooled LMFRs, there was the Super-Phénix 1 in France [26], commissioned in 1986 and decommissioned in 1997.

The structural materials used in contact with molten Na have been varying stainless steels, mainly 316, and those containing higher concentrations of Cr and Ni [26]. Studies have been conducted to observe the interaction of select stainless steels with liquid Na. One study, performed by Suzuki et al. [28], explored the reactivity of numerous stainless steel alloys, including 316SS, by flowing molten Na at a rate of 4 m/s at 700 °C and evaluating the specimens after 426, 1723, 2395 and 4066 h. They found that for all alloys, Cr and Ni were selectively dissolved. The decrease in the

concentration of these elements was initially rapid, but began to level off after 2000 h, and, after 4000 h, the concentration of Cr and Ni became stable.

Optical microscopy, OM, of the cross sections showed the formation of the σ -phase (a chromium/molybdenum-rich intermetallic phase) [29] to be approximately 50 μm from the surface in 316SS. All alloys tested except for a low Mo content alloy formed small, surface corrosion nodes rich in Mo and Fe. The attack by Na on some of the alloys was determined to be due to their higher Ni content, which showed signs of intergranular attack of more than 10 μm , as well as pores of less than 10 μm on the surface. Another study, conducted by Ganesan et al. [30], explored the reactivity between annealed 316 stainless steels and static Na at 773 and 873 K for durations ranging from 500 to 2000 h in an Ar atmosphere. Again, it was found that Fe-Mo corrosion resistant nodes were formed on the exposed surfaces. Ganesan et al. [30] also found the post-exposure weight losses for the annealed samples, under static Na, were less than those reported in dynamic Na environments [31]. The weight loss in the annealed samples was also seen to be less than the 20 % found in cold worked 316 stainless steel samples [32,33]. As with Suzuki et al. [28], the σ -phase was found to nucleate near the exposed surfaces. A increase in micro-hardness - from 150 ± 7 V_H to 250 ± 10 after exposure to Na - was also noted.[30] Investigations into the use of coatings, such as conversion coating of perovskites,[34] or the formation of Si diffusion layers,[35] on the surface of stainless steels have been found to have some success in retarding corrosion. Another possible solution for coatings could be the spray coating of the MAX phases.[36-38]

Herein, we focus on the interactions of four MAX phases, Ti_3SiC_2 , Ti_2AlC , Ti_3AlC_2 and Cr_2AlC with static molten Na held at 550 °C or 750 °C for 168 h (one week).

II. Experimental Procedure

The Ti_2AlC samples were prepared by loading pre-reacted Ti_2AlC powders (Kanthal, Hallstahammar, Sweden) in a graphite die and hot pressing (HPing) at a rate of $500\text{ }^\circ\text{C/hr}$ to $1300\text{ }^\circ\text{C}$. The hold time at that temperature was 4 h under a load corresponding to a stress of $\approx 30\text{ MPa}$ and a vacuum of 10^{-2} torr.

The Ti_3AlC_2 samples were prepared by HPing pre-reacted Ti_2AlC (Kanthal, Hallstahammar, Sweden) and TiC (Alfa Aesar, Ward Hill, MA) in a 1:1 mole ratio to obtain the 3:1:2 stoichiometry of $\text{Ti}:\text{Al}:\text{C}$. The powders were weighed, mixed, ball milled for 24 h, placed in a graphite die, and heated in a graphite-heated HP under a vacuum of 10^{-2} Torr at a rate of $500\text{ }^\circ\text{C/h}$ to $1400\text{ }^\circ\text{C}$. The sample was held for 4 h at this temperature under a pressure of $\sim 40\text{ MPa}$ before cooling.

The coarse-grained polycrystalline Ti_3SiC_2 samples were prepared by ball milling Ti , SiC , and graphite powders (all from Alfa Aesar, Ward Hill, MA) and cold pressing under 180 MPa into bars. These bars were then hot isostatically pressed, HIPed, at $1600\text{ }^\circ\text{C}$ under a pressure of $\sim 40\text{ MPa}$ for 4 h to produce a coarse-grained microstructure.

The Cr_2AlC samples were prepared by ball milling a 2:1:1 stoichiometric mixture, respectively, of Cr , Al and graphite powders (all from Alfa Aesar, Ward Hill, MA) for 24 h. This mixture was then poured into a graphite mold and HPed at $1400\text{ }^\circ\text{C}$ under a pressure of $\sim 40\text{ MPa}$ for 4 h under a vacuum of $\approx 1\text{ Pa}$.

The fully dense samples were cut using a diamond saw into $8 \times 25 \times 2\text{ mm}^3$ bars. These bars, along with Na pieces (99.95%, Alfa Aesar, Ward Hill, MA) were placed in 316 stainless tubes with outer diameter 1.6 cm, length $\approx 7.6\text{ cm}$ and a 0.2 cm wall thickness. Swagelok caps were used to seal both ends of the tubes and to create an

airtight seal. In order to limit the amount of oxygen present in the tubes, a backflow of Ar gas was used during the sealing process. The sealed tubes were in turn placed in a box furnace and heated at a rate of 10 °C/min to either 550 °C or 750 °C for a week, or 168 h, before furnace cooling.

Upon cooling to room temperature, the samples were cross-sectioned from the tubes using a hacksaw, the excess Na was gently scraped off, and the samples were mounted in epoxy. The mounted samples were then ground and polished in water in preparation for observation in a scanning electron microscope (SEM), (FEI Zeiss Supra 50VP). Note that the use of water here dissolves any excess Na.

X-ray diffraction (XRD) patterns were obtained, using a diffractometer (Rigaku SmartLab, Tokyo, Japan) with a 5 mm or 2 mm slit, depending on sample size limitations. The scans were conducted over the range of 5 to 70° 2 θ , with a step size of 0.04°. The scans were performed on one of the surfaces exposed to Na at 750 °C after removing the samples from the stainless steel tubes. In this case, no water was used, and, therefore, any water-soluble reaction phases that may have formed would be captured in the XRD patterns.

An optical microscope, OM, was used to view the etched surface of each sample. The etchant used for the Ti₂AlC, Ti₃AlC₂, and Ti₃SiC₂ samples was a 1:1:1 part solution of hydrofluoric acid (48%, Sigma Aldrich, St. Louis, MO), nitric acid (68%, Alfa Aesar, Ward Hill, MA) and water. A drop of etchant was placed on the surface of each sample and rinsed after 2-3 s. For the Cr₂AlC sample, a drop of hydrofluoric acid (48%, Sigma Aldrich, St. Louis, MO) was placed on the sample's surface and rinsed with water after ~ 120 s.

Transmission electron microscope (TEM) lamellae of the MAX samples were prepared using a focused ion beam (FIB) via a typical in situ lift-out procedure on a FEI

Strata DB235 dual-beam FIB (Hillsboro, OR). Cross-sectional TEM analysis was performed on a JEOL JEM2100 (Tokyo, Japan) LaB₆ equipped with a high-resolution objective-lens pole piece.

Micro-hardness measurements were carried out on a hardness tester (LECO M-400, St. Joseph, MI) at 1 kg force. At least ten indentations were made on each of the unreacted cross-sections, reacted cross-sections, and reacted surfaces (i.e. surfaces exposed to Na) of all four MAX compositions.

III. Results and Discussion

Since the XRD diffraction patterns before (not shown) and after (Fig. 1) Na exposure at 750 °C were identical, it is reasonable to conclude that no crystalline phases formed as a result of the exposure. Percentage of TiC in the as-prepared Ti₂AlC, Ti₃AlC₂, and Ti₃SiC₂ samples were 0.3(3) wt.%, 1.2(3) wt.%, and 22(1) wt.%, respectively, while Cr₂AlC contained 0.3(2) wt.% of the binary phase, Cr₇C₃. These percentages were unchanged after exposure. Note that the post-Na exposure XRD patterns were taken on surfaces that were never exposed to water. The remaining questions are: did any amorphous or nanocrystalline phases form and/or was there any evidence for grain boundary attack? We note in passing that the samples exposed to Na at 550 °C fared better than those at 750 °C and are not discussed further. In fact, it was the apparent lack of a reaction at 550 °C that prompted us to increase the exposure temperature to 750 °C.

Figure 1: Bulk XRD patterns of surfaces exposed to Na at 750 °C for 168 h of, a) Ti_2AlC , b) Ti_3AlC_2 , c) Ti_3SiC_2 , and d) Cr_2AlC . Red squares represent the MAX phases; blue circles represent the binary carbides that were the major impurities found in the as-fabricated.

Figures 2a,b,c,d, 2e,f,g,h, 3a,b,c,d and 3e,f,g,h, show OM and SEM micrographs pertaining to Ti_3SiC_2 , Cr_2AlC , Ti_2AlC and Ti_3AlC_2 , respectively. In all cases, panels a) and e) show an etched OM micrograph after exposure to Na at 750 °C for a week. Panels b) and f) show a secondary electrons, SE, SEM micrograph of the as-fabricated sample. Panels c), d), g), and h) show SE and backscattered electron, BSE, SEM micrographs of the same samples, respectively. In all micrographs, the emphasis was on the corners of the samples, since the latter typically experience the highest stresses when reaction layers or grain boundaries are attacked. The reason that micrographs of the samples before testing are shown is to emphasize that the corners were not sharp initially. There was also no noticeable changes were seen in weight gain/loss

measurements. Said otherwise, the fact that the corners in the exposed samples were not sharp is not due to Na attack. Note the change of scale in Figs. 3f, g, and h)

Figure 2: Micrographs of Ti_3SiC_2 , a) reacted and etched surface, OM; b) unreacted, SE SEM; c) reacted, SE SEM; d) reacted, BS SEM, and Cr_2AlC , e) reacted and etched surface, OM; f) unreacted, SE SEM; g) reacted, SE SEM; and, h) reacted, BS SEM, respectively, after contact with Na at 750 °C for 168 h.

Based on the totality of our results, and consistent with the XRD results, it is fair to conclude that no *reaction layers*, such as any new phases, formed as a result of the exposure.

The next question is: were the grain boundaries attacked? When the micrographs of the as-received and exposed samples are compared for Ti_3SiC_2 (compare Figs. 2b, with 2c and d), Cr_2AlC (compare Figs. 2f with 2g and h) and Ti_2AlC (compare Figs. 3b with 3c and d) it does not appear that the grain boundaries were attacked. This

conclusion is bolstered by our micro-hardness measurements on the cross-sections and exposed surfaces of these compositions shown in Table 1. Given that the V_H changes little before and after Na-exposure is consistent with the lack of grain boundary attack. It follows that not only are the Ti_3SiC_2 , Cr_2AlC , and Ti_2AlC compositions apparently stable vis-à-vis exposure to molten Na, but also their grain boundaries.

Figure 3: Micrographs of Ti_2AlC , a) reacted and etched surface, OM; b) unreacted, SE SEM; c) reacted, SE SEM; d) reacted, BS SEM, and Ti_3AlC_2 a) reacted and etched surface, OM; e) unreacted, SE SEM; f) reacted, SE SEM; and, g) reacted, BS SEM, respectively, after contact with Na at 750 °C for 168 h.

Evidence for grain boundary attack in Ti_3AlC_2 is best seen in Fig. 3e, g and h. In the OM micrograph (Fig. 3e), the colours of the grains near the edges are slightly darker than from those in the bulk. In Fig. 3h, EDS of the dark regions between the grains showed high levels of C. This high C signal is most probably associated with epoxy that penetrated between the grains during sample mounting. The thickness of the

affected layer is about 50 μm . Note that no Na signal was found in any of the micrographs, however.

A FIB liftout was taken from an area within 50 μm from the $\text{Ti}_3\text{AlC}_2/\text{Na}$ interface of a sample held at 750 $^{\circ}\text{C}$ for 168 h. The liftout contained two different grains (Fig. 4). Contrast at the grain boundary suggests the presence of dislocations, best seen in Fig. 4a. EDS results taken across the two grains and the interface showed a Ti/Al ratio of more than 3. Interestingly, a greater deficiency of Al was found inside the grain (Ti/Al ratio of 5.0(3) and 4.4(1) for grains 1 and 2, respectively) than at the grain boundary (Ti/Al ratio of 3.8(6); value in parentheses represents the uncertainty on the last significant digit, taken as one standard of deviation). These results suggest a migration of Al from the grain to the grain boundaries, and out into the Na.

A TEM sample of the other Ti-Al MAX phase, Ti_2AlC , taken from the Na/MAX interface, showed no change in Ti/Al ratios across grains or grain boundaries, with the ratio remaining constant at 2.

Figure 4: TEM micrographs of Ti_3AlC_2 after contact with Na at 750 °C for 168 h, a) with objective aperture, and, b), normal bright field with diffraction patterns for grains 1 and 2.

Here again, the V_H results support the conclusion that a reaction of sorts occurred between Ti_3AlC_2 and the molten salt. V_H decreased from 480 ± 5 on the unreacted cross section to 441 ± 3 on the reacted cross section. At 399 ± 5 , the V_H values for surfaces exposed to Na were even lower. This reduction in hardness is most probably related to grain boundary attack. Another reason could be the reduction of the Al concentration in the bulk of the grains. Which mechanism is operative is beyond the scope of this work. Further work is needed to better understand whether this attack is intrinsic or extrinsic. Given that the grain boundaries in the compositionally related Ti_2AlC samples were not attacked, it is more likely than not that the attack is intrinsic.

Table 1: Vickers Hardness values (V_H) at 1 kg force of unreacted cross-section, reacted cross-section, and reacted surface of MAX phases tested herein. Values in parentheses represent one standard deviation.

Vickers Hardness	Ti₃SiC₂	Cr₂AlC	Ti₂AlC	Ti₃AlC₂
Unreacted Cross-section	440(10)	466(8)	458(6)	480(5)
Reacted Cross-section	455(9)	451(5)	467(5)	441(3)
Reacted Surface	450(10)	476(8)	458(2)	399(5)

IV. Summary and Conclusion

The interactions between polycrystalline samples of Ti₂AlC, Ti₃AlC₂, Ti₃SiC₂ and Cr₂AlC, and pure, static Na at 550 °C and 750 °C for 168 h was studied for the first time. The MAX samples were placed in sealed stainless steel tubes. Based on a comparison of pre- and post-XRD, OM, SEM and TEM, micrographs, EDS, and Vickers microhardness measurements, we conclude that not only are the Ti₃SiC₂, Cr₂AlC and Ti₂AlC compositions stable vis-à-vis exposure to molten Na, but also their grain boundaries. In contradistinction, the evidence shows that both the bulk of the Ti₃AlC₂ grains and the grain boundaries are altered by the molten Na. TEM and EDS results suggest a migration of Al to the grain boundaries, and into the molten Na.

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