

Corrosion Behavior of V_2AlC and Cr_2AlC Materials in Acidic Media

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Received on:23/10/2014 & Accepted on:2/4/2015

Abstract

This work involves the manufacturing of MAX phase materials include V_2AlC and Cr_2AlC using powder metallurgy as a new class of materials which characterized by regular crystals in lattice. Corrosion behavior of these materials was investigated by Potentiostat to estimate corrosion resistance and compared with the most resistant material represented by SS 316L. The experiments were carried out in 0.01N of HCl and 0.01N H_2SO_4 at four temperatures in the range of 303-333K. Polarization resistance values which calculated by Stern-Geary equation indicate that the MAX phase materials more resistance than SS 316L. Optical microscopy results for corroded surfaces were confirming the resistivity of MAX phase materials.

Key word: MAX phase materials, Corrosion behavior, V_2AlC , Cr_2AlC .

السلوك التآكلي للمواد V_2AlC و Cr_2AlC في أوساط حامضية

الخلاصة

يتضمن هذا البحث تصنيع مواد الطور MAX والتي تتضمن كلا من V_2AlC و Cr_2AlC باستخدام ميتالورجيا المساحيق كمواد جديدة تتميز بانتظام البلورات في الشبكة البلورية. تم التحري عن السلوك التآكلي لهذه المواد بالمجهاد الساكن لتقييم مقاومة التآكل ومقارنتها مع الفولاذ المقاوم للصدأ 316L الذي يعد من أكثر المواد مقاومة للتآكل، اجري فحص التآكل في حامض الهيدروكلوريك والكبريتيك بتركيز 0.01 مولالي وبمدى من درجات الحرارة تتراوح بين 303 و 333 كلفن. حسب قيم مقاومة التآكل وفقاً لمعادلة ستيرن-جيري وبينت بان مقاومة المواد المحضرة أكثر من مقاومة الفولاذ المقاوم للصدأ وكانت هذه النتائج متوافقة مع نتائج فحص التصوير البصري للمواد المحضرة.

كلمات مرشدة: مواد الطور MAX، السلوك التآكلي، V_2AlC ، Cr_2AlC .

INTRODUCTION

It's known that the structure and composition of both metals and alloys are important in deciding their corrosion characteristics. Indeed, structure and composition are critical in many forms of localized corrosion. For a metal or alloy to corrode evenly, the anodic and cathodic sites must be interchangeable. This implies that every site on the surface is energetically equivalent and therefore equally susceptible to dissolution, but this is never the case [1]. Acids can be either oxidizing or reducing in nature. Some metals are resistant to oxidizing acids (e.g. nitric) while others are resistant to reducing acids (e.g. hydrochloric or sulfuric). By alloying such metals it is possible to produce materials that resist corrosion in both media. The choice of an alloy for a specific environment will depend on the acid or the mixture of acids present, on concentration, temperature, aeration, contaminants, flow characteristics, the presence and tightness of crevices, other material in the system, and many other environmental conditions.

The search for materials which have the combination of both metal and ceramic properties is becoming more important in the 21st century [2]. The MAX phase materials are made up of an early transition metal M in the periodic table, an element from the A groups, usually IIIA and IVA, and a third element, X, which is either nitrogen or carbon, in the composition $M_{n+1}AX_n$, where n is 1, 2 or 3.

Many authors were interested fabrication of MAX phase materials and studied some of their physical and mechanical properties. Zhou et al. in 2001 determined the electronic and structural properties of the layered ternary compound Ti_3AlC_2 using the *ab initio* pseudopotential method based on density functional theory [3]. Wang and Zhou investigated the isothermal oxidation behavior of Ti_2AlC at intermediate temperatures of 500, 600, 700, 800, and 900°C in flowing air by means of thermogravimetric analysis, X-ray diffraction (XRD), Raman spectroscopy, and scanning electron microscopy (SEM)/energy dispersive spectroscopy [4]. They also fabricated the microstructure of polycrystalline Ti_3AlC_2 by the solid-liquid reaction synthesis and simultaneous in situ hot pressing process was investigated by SEM and TEM [5].

Sun et al. performed theoretical studies of the bulk modulus of M_2AlC , where $M = Ti, V, Cr$ by means of *ab initio* total energy calculations using the projector augmented wave methods. The bulk modulus of M_2AlC increases as Ti is substituted with V and Cr by 19% and 36%, respectively. This can be understood since the substitution of Ti by V and Cr is associated with an extensive increase in the M–Al and M–C bond energy [6].

Schneider et al. calculated the equilibrium volume and the density of states (DOS) of Cr_2AlC for antiferromagnetic (AFM), ferromagnetic (FM) and paramagnetic (PM) configurations by *ab initio* total energy calculations [7]. Hettinger et al. investigated the electronic, magnetotransport, thermoelectric, thermal, and elastic properties of four M_2AlC phases: Ti_2AlC , V_2AlC , Cr_2AlC and Nb_2AlC . The electrical conductivity, Hall coefficient, and magnetoresistances are analyzed within a two-band framework assuming a temperature-independent charge carrier concentration [8]. Khoptiar et al. synthesized a near-single-phase Ti_3AlC_2 ternary carbide from 3Ti–1.1Al–1.8C powder blend, both by the wave propagation and thermal explosion (TE) modes of self-propagating high temperature synthesis [9]. Sun et al. studied M_2AlN phases, where $M = Ti, V, Cr$, by means of *ab initio* total energy calculations [10]. Tian et al. fabricated Cr_2AlC ceramics by hot-pressing using Cr, Al

and C powders as starting materials. The phase assemblages of the samples consisted of Cr₂AlC, as a major crystalline phase, together with a very small amount of Cr₇C₃ and an unknown phase [11]. Tian et al. fabricated dense and predominantly single-phase samples of Cr₂AlC, together with a trace amount of Cr₇C₃ by hot pressing of a mixture of chromium, aluminum, and graphite powders at 1400°C for 1 h. The material exhibits good damage tolerance [12]. Music et al. studied the electronic structure of Ta_{n+1}AlC_n (space group *P63/mmc*, *n* = 1–3) under uniform compression from 0 to 60 GPa and at temperatures from 0 to 1500 K using *ab initio* calculations [13]. Zhou et al. synthesized new layered compounds, (V_{0.5}Cr_{0.5})₃AlC₂, (V_{0.5}Cr_{0.5})₄AlC₃, and (V_{0.5}Cr_{0.5})₅Al₂C₃ by reactive hot pressing V, Cr, Al, and graphite powders [14]. Tian et al. studied the isothermal oxidation behavior of Cr₂AlC ceramics oxidized in air at 1100 and 1250 °C for 20 h [15].

Also they studied the compressive properties of ternary compound Cr₂AlC at different temperatures and strain rates [16]. Zhou et al. synthesized dense bulk Cr₂AlC by hot-pressing of Cr, Cr₃C₂ and Al powders as starting materials [17]. Kim et al. studied the Cr₂AlC ternary carbide in chemical and oxidation resistances as well as mechanical properties at high temperatures [18]. Many authors studied the corrosion of metals and alloys in HCl [19-21] and H₂SO₄ [22,23], also for composite materials [24-27] but there are little researches concerned the corrosion behavior of MAX phase materials in acidic medium.

The aim of present work is fabricated the V₂AlC and Cr₂AlC materials by powder metallurgy and study their corrosion behavior in acidic media include HCl and H₂SO₄ with 0.01N concentration and comparison their behavior with Stainless Steel 316L which acts as most metallic resistant alloys at four temperatures 303, 313, 323 and 333K. The importance of present research is to study the corrosion behavior of new class of material which refers to MAX phase materials to use it as reproducible industrial materials.

EXPERIMENTAL PROCEDURE

To fabricate the V₂AlC and Cr₂AlC samples V, Cr, C, and Al powders (99% pure) were mixed in stoichiometric proportions, ball milled (BAIRD & TATLOCK) for 20 min at high level of speed for each sample, cold pressed using the hydraulic press machine type (Mega 50 Ton Max) and placed in a graphite die in a vacuum hot press (MTI Corporation GLS 1500X). The latter was evacuated and heated to 1100-1350 °C for 6 h. The sample was held at the maximum applied uniaxial pressure ~1.7 MPa for 10 min.

To characterize the prepared MAX phase material, X-ray Diffraction (XRD) analysis was used in order to find out the composition and phase identification of each sample using Shimadzu X-ray diffractometer (type XRD- 6000/7000). Electrochemical measurements were performed with a potentiostat by SCI electrochemical software at a scan rate 5 mV.sec⁻¹. Polarization experiments were started when the rate at which open circuit potential (E_{ocp}) changed was less and more 300mV. The main results obtained were expressed in terms of the corrosion potentials (E_{corr}) and corrosion current density (i_{corr}) in addition to measure the Tafel slopes by Tafel extrapolation method. From the values of Tafel slopes and corrosion current density, the polarization resistances values can be calculated according to Stern-Geary equation.

Acidic medias in this work were 0.01N HCl solution (pH=2) and 0.01N H₂SO₄ solution (pH=2.3) which used for corrosion tests. All experiments were achieved at

four temperatures which adjusted by water bath. The microstructure evolution was investigated by means of optical microscope using (BEL photonics) microscope was connected to computer.

RESULTS AND DISCUSSION

The X-ray powder diffraction patterns collected at 1 atm for V₂AlC and Cr₂AlC are shown in Fig. (1). For two materials, all major peaks were assigned to the hexagonal structure with the space group *P63/mmc*. A few low intensity impurity peaks were not identified. XRD test shows good agreement with observed test by Bouchaib [28].

Figure (2) shows the Tafel plots of V₂AlC, Cr₂AlC in 0.01N HCl solution at four different temperatures. The plot shows the active region representing by cathodic and anodic sites. At cathodic sites, reduction of hydrogen ions to molecules takes place as follow:



While at anodic sites, dissolution of metals can occur. From the active region, the corrosion parameters can be calculated by Tafel extrapolation method to estimate the corrosion behavior of material. Hydrochloric acid considers the most corrosive environment due to high acidity and chloride ions which able to destroy any passive film may be formed on the metal surface. It is clear from the polarization curves that V₂AlC material gave shifting to lower current values compared with Cr₂AlC. On the other hand, Cr₂AlC material shows the formation of passive film which makes it more resistant material to corrosion in HCl solution.

The data of corrosion are listed in Table (1) which indicates that the lowest corrosion current densities were for V₂AlC and take the following sequence:

$$i_{\text{corr}} \text{ in } 0.01\text{N HCl} \quad \text{V}_2\text{AlC} < \text{Cr}_2\text{AlC} < \text{SS 316L}$$

The polarization resistance (R_p) may be defined as the slope of a potential (ΔE)-current density (Δi). The term (R_p) corresponds to the resistance (R) of the metal/solution interface to charge –transfer reaction. It is also a measure of the resistance of the metal to corrosion in the solution in which the metal is immersed. The polarization resistance (R_p) can be determined from Stern- Geary equation [29]:

$$R_p = \left(\frac{dE}{di} \right)_{i=0} = \frac{b_a * b_c}{2.303 * i_{\text{corr}} * (b_a + b_c)} \quad \dots(2)$$

Where

b_c and b_a are cathodic and anodic Tafel slop respectively. The values of R_p are presented in Table (1). These data indicate that the polarization resistance was good agreement with the results of corrosion current density.

The corrosion current density (i_{corr}) is a kinetic parameter and represents the rate of corrosion under specified equilibrium condition. Any factor that enhances the value of (i_{corr}) results in an enhanced value of the corrosion rate on pure kinetic ground.

Sulphuric acid is another example of corrosive environment due to sulphate ions which may be form soluble salts with metals. The data of Table (2) indicate that corrosion potentials of SS 316L are more noble than these for V₂AlC and Cr₂AlC materials, the corrosion potential (E_{corr}) of a material in a certain medium at a

constant temperature is a thermodynamic parameter which is a criterion for the extent of the corrosion feasibility under the equilibrium potential.

The data of Figure (3) and Table (2) indicate the corrosion current densities in 0.01N sulphuric acid which take the following sequence:

i_{corr} in 0.01N H₂SO₄ V₂AlC < Cr₂AlC < SS 316L

The data of polarization resistance enhanced the results of corrosion current densities, i.e., the corrosion behavior of MAX phase materials better than SS 316L due to regular crystals in structures.

Optical microscopies enhanced the resistivity of MAX phase materials to corrosion in 0.01N HCl and 0.01N H₂SO₄ media. Figures (4) and (5) show the polished surfaces of V₂AlC and Cr₂AlC materials respectively at two magnifies. These figures indicate the homogenous surface for both MAX materials. The microstructure test of Cr₂AlC shows the formation of this phase, in addition to form Cr₂C₃ (dark region) and Cr₇C₃ phase can be found (white region) [30]. From Figures (6) and (7), little changes have been noticed on the corroded surfaces of MAX materials which enhanced the low corrosion damage for these materials.

In many engineering structures, dissimilar alloys are connected in a variety of ways, crevices often exist, and the environment is variable and dependent on local flow conditions. Furthermore, metals and alloys are typically covered with a surface layer. Their corrosion behavior is influenced by the surface layer and by a host of metallurgical factors. Even extremely pure single crystals have defects that can affect corrosion, but impurities and alloying elements, grain boundaries, second phases, and inclusions often have a dominant effect [31].

In general, the most susceptible site or defect on a metal surface will be the first to be attacked when it is exposed to a corrosive environment. Sometimes such attack simply results in innocuous removal of the susceptible material, leaving a surface with improved corrosion resistance. The most susceptible defects that lead to sustained attack will control the form of corrosion. The exposure of structural and compositional inhomogeneities to corrosive environments invites the possibility of enhanced or preferential (localized) attack. This fact is used to great advantage in metallographic analysis, where chemical etching is frequently used to reveal the microstructural characteristics of a material for the optical microscope, but is usually a negative attribute in materials applications.

In Crystallography and defects, corrosion results in the removal of atoms from a metal by dissolution or conversion to an oxidized phase, such as an oxide or sulfide. The metal atoms most likely to undergo corrosion are those with the highest free energy. Thus, atoms located within the bulk of the material are much less susceptible to corrosion than those in the outermost layers of the metal surface, and corrosion reactions are generally considered to be surface chemistry [31].

Conclusion

From the study of corrosion behavior for MAX phase materials can be concluded that these materials have good corrosion resistance in acidic media. This conclusion was confirmed through the polarization test and optical microscopy due to homogeneously structures and regular crystal in lattice of these ceramic materials.

Table (1): Corrosion parameters for V₂AlC, Cr₂AlC and SS316L in 0.01N HCl at four temperatures.

Material	Temp. K	-E _{corr} mV	i _{corr} μA.cm ⁻²	-b _c mV.dec ⁻¹	+b _a mV.dec ⁻¹	R _p x10 ³ Ω.cm ²
V ₂ AlC	303	564.1	70.09	118.4	102.1	0.33964
	313	575.5	84.29	150.7	116.7	0.338807
	323	585.8	98.15	116.6	110.4	0.250875
	333	577.7	116.83	99.2	106.7	0.191061
Cr ₂ AlC	303	732.4	125.14	94.4	133.9	0.192113
	313	723.8	155.55	110.6	185.5	0.193418
	323	724.6	172.49	107.6	184.2	0.170985
	333	732.1	203.81	100.2	196.9	0.141479
SS 316L	303	532.9	115.75	86.5	73.8	0.149391
	313	542.6	341.79	133.8	126.9	0.082742
	323	547.2	367.19	149.4	106.7	0.073607
	333	563.0	452.17	165.1	138.8	0.072412

Table (2): Corrosion parameters for V₂AlC, Cr₂AlC and SS316L in 0.01N H₂SO₄ at four temperatures.

Material	Temp. K	-E _{corr} mV	i _{corr} μA.cm ⁻²	-b _c mV.dec ⁻¹	+b _a mV.dec ⁻¹	R _p x10 ³ Ω.cm ²
V ₂ AlC	303	581.1	48.54	141.3	117.5	0.573881
	313	610.8	49.39	114.8	114.3	0.503535
	323	623.8	49.74	95.1	99.3	0.424066
	333	617.9	61.54	94.3	91.5	0.327669
Cr ₂ AlC	303	727.6	62.55	75.3	112.9	0.31358
	313	738.5	68.45	150.2	147.8	0.472564
	323	747.0	70.15	154.3	173.7	0.50579
	333	747.1	74.16	161.4	167.4	0.481131
SS 316L	303	518.0	104.27	95.5	103.9	0.207224
	313	514.2	143.00	111.9	113.0	0.170722
	323	517.0	153.41	99.3	105.0	0.144452
	333	513.0	164.51	94.7	98.7	0.127563

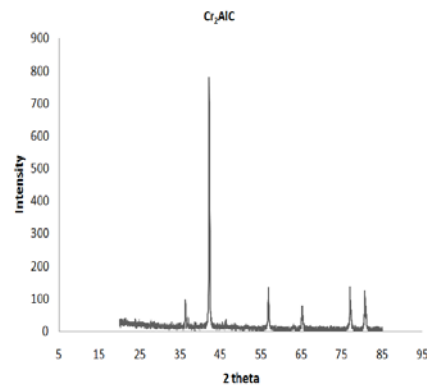
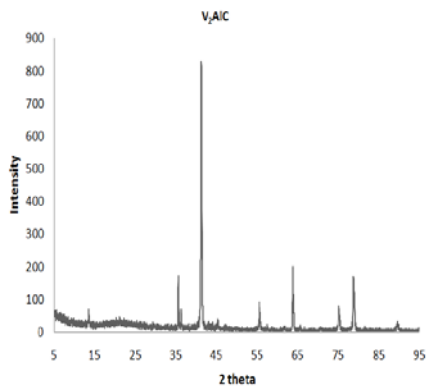


Figure (1): XRD for prepared MAX material.

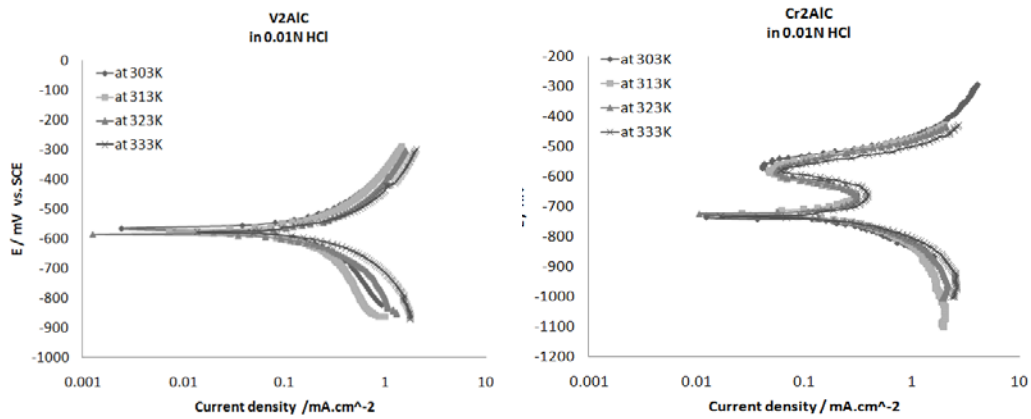
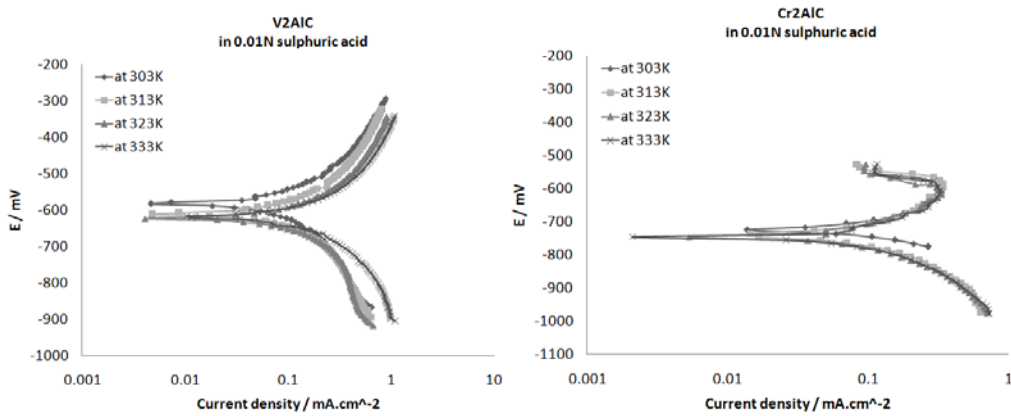
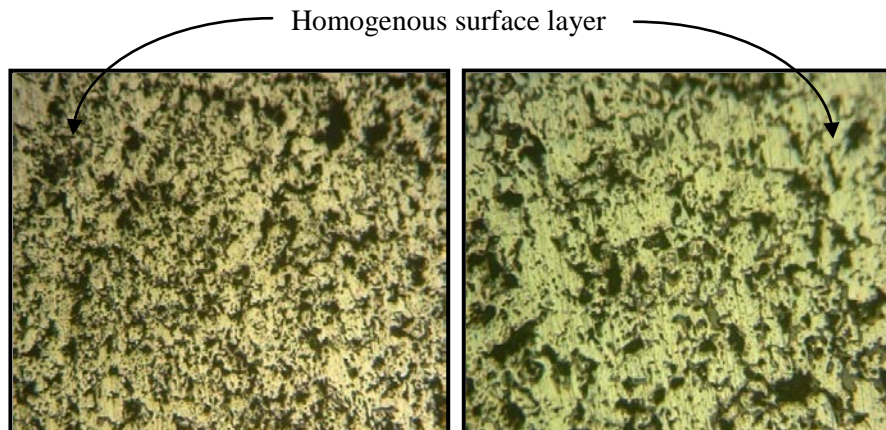


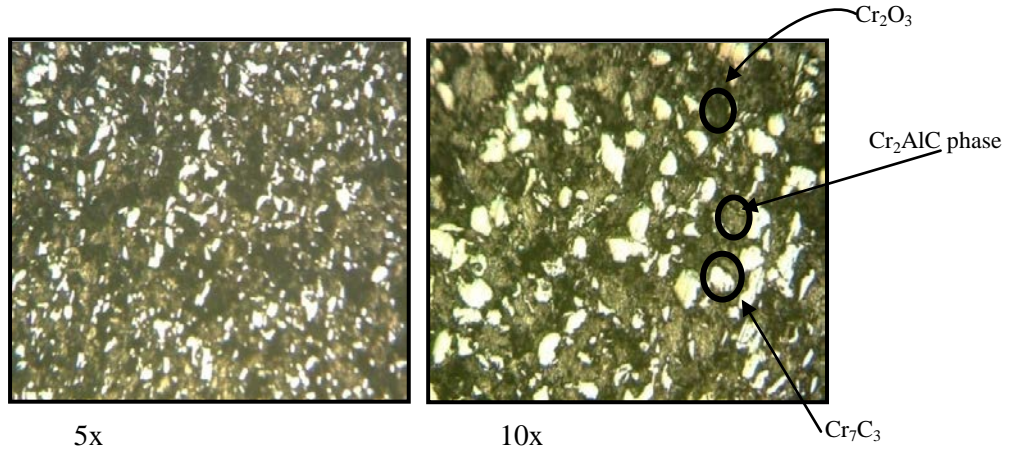
Figure (2): Tafel plot of MAX phase materials in 0.01N HCl at four temperatures.



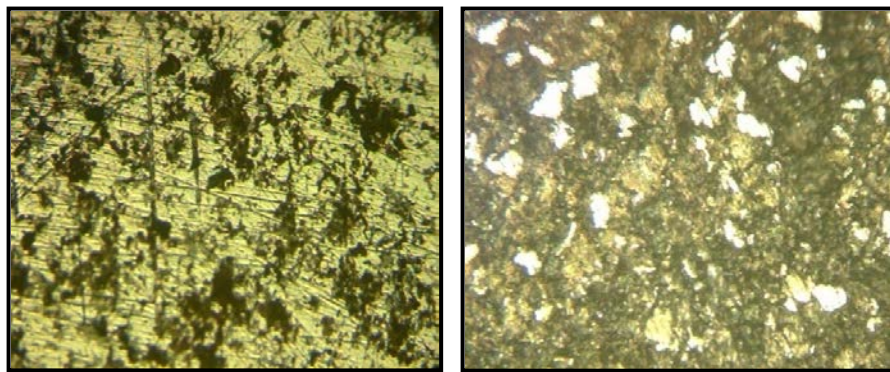
Figure(3): Tafel plot of MAX phase materials in 0.01N H₂SO₄ at four temperatures.



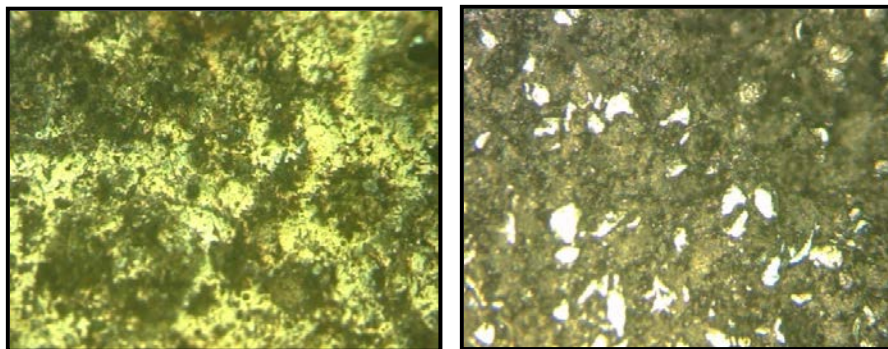
5x 10x
Figure(4): Optical microstructure for polished surface of V_2AlC material at two magnifies.



5x 10x
Figure(5): Optical microstructure for polished surface of Cr_2AlC material at two magnifies.



V_2AlC Cr_2AlC
Figure(6): Optical microstructure for corroded surfaces of MAX phases material 0.01N HCl medium at 10X.



V_2AlC

Cr_2AlC

Figure (7): Optical microstructure for corroded surfaces of MAX phases material $0.01N H_2SO_4$ medium at 10X.

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