Improved of the Nanolaminate - Ceramic by the Additional of Aluminium to used in Higher Resistance Hydraulic Structures

Jawad Kadhim Abbood **Jabbar Abbas Jaber**
Mussaib Technical Institute - Babylon
Wisam Abidalabbas Abidalla **Mohammed Ali Nasser**
Mussaib Technical Institute - Babylon *Petrol Dist. Babylon*
r_algasbaw@yahoo.com

Abstract :-

It has been tried in this research to introduce this type of new structural materials as reproducible industrial materials and overtake some production obstacles by exploring the direction of chemistry structure relation. The compacted samples have been prepared from elemental powders (Titanium, Aluminum and Black Carbon) using the powder technology techniques. They have been dispersed in different concentrations (Ti-Al-C) and different molar ratios due to the change in the Ti to Al concentrations. Thus the effect of Al concentration on MAX phase evolution in the Ti-Al-C system has been investigated and discussed. Therefore, the concentration of Al has improved the applications of water resource structures, and that alloy, especially in corrosion resistance and under water, has proved structures as well as pipes because the Al has created the layer that protects this ceramic from hard and nature conditions , So the Al metal lead to use this ceramic in loading bearing structures such as bridges and high impact resistance such as Dams. The effects of cold and hot pressing as well as sintering in different temperatures are investigated. Phase evolution is discovered by X-ray diffraction (XRD), and SEM. Micro hardness test and Archimedes method is used to measure the density and porosity percentage. No evidence for the direct formation of MAX phases from elemental powders is foud which may explain the need of high temperatures to produce such phases since the breaking of bonds is required for the intermediate phase.

Key word : Aluminium , Nanolaminate - Ceramic , Higher Resistance Hydraulic Structures

الخلاصة

جرت محاولة في هذا البحث لتقديم منتج إنشائي جديد كنوع من المواد الصناعية الثانوية مثل السيراميك والتي تقاوم تأثير المياه وتتجاوز بعض المنتجات المعقدة وذلك بواسطة (اتجاه الربط) بالتركيب الكيمياوي . النماذج المكبوسة تحضر من مساحيق العناصر (التيتانيوم ، الألمنيوم، الكاربون الأسود) والتي تم تشكيلها بتراكيز مختلفة ومختارة باستخدام تقنية استخدام تكنولوجيا المساحيق وبنسب مولارية اعتمادا على تغيير نسبة تراكيز عنصر التيتانيوم إلى عنصر الالمنيوم لذلك فان تأثير تركيز الالمنيوم على تطوير الطور الأحادي لخلط العناصر قد تم التحري عنها ومناقشتها .

تبين إن تراكيز الالمنيوم قد حسنت من استخدام المنتج في المشاريع الكبيرة والمهمة كالمنشات الخاصة بالموارد المائية، خصوصا في مجال مقاومة التآكل والمنشات المانعة للتسرب تحت الماء للمنتج بسبب تكوين الالمنيوم طبقات حماية تقاوم الظروف الطبيعية والصعبة، وعليه يمكن استخدامه في المنشات ذات التحمل العالي مثل الجسور والمنشات التي تحت تأثير الصدمات والتحمل العاليين مثل السدود. تأثير الضغط البارد والحار والتلبيد بدرجات حرارة مختلفة تم التحري عنها. تم استكشاف سلوك الأطوار الأحادية بواسطة أشعة اكس وإجراء فحص المخطط المجهري الالكتروني، فحص قوة الصلادة المجهري وكذلك فحص طريقة (ارخميدس) لقياس نسب الكثافة والمسامية . تبين انه ليس من السهولة الحصول على الطور الأحادي مباشرة من خلال العناصر التي تحتاج إلى درجات حرارة عالية لإنتاج الطور الأحادي بعد كسر الأواصر بين الأطوار الوسطية .

الكلمات المفتاحية : الألمنيوم ، النانوسيراميك ، المنشات الهيدروليكية المائية ذات المقاومة العالية .

Introduction :-

Ternary ceramics are compound comprising covalent, metallic and ionic bonds in one crystal cell, which result in unusual properties combining the merits of both metals and ceramics. Like metals, they are thermal and electrical conductive, easy to be machined with conventional tools and resistant to thermal shock; like ceramics they present a high strength, high melting point and high thermal stability [Ahmed,2011;Alany,1999].The ternary ceramics (carbides or nitrides) are three-component systems that are similar to two-component systems except that there are four independent variables: pressure, time, temperature and the concentration of two components (which fix the third). If pressure is arbitrarily fixed, the presence of four phases gives rise to an invariant system[Daniel,2007;Zhou,2007,Bouchaib,2006]. This combination of properties makes ternary ceramics interesting for high temperature and high impact resistance applications loading bearing structures besides considering as the first choice for many potential applications. Polycrystalline ternary ceramics have previously been synthesized primarily as bulk material by reactive hot isostatic pressing [Lin *et. al*.,2005;Tian *et. al*.,2007; Zhijun *et. al.* ,2007]. This may give the ability to produce ternary ceramics in different shapes. ternary ceramics have been indicated a high thermal stability bulk materials[Maurice,2005]. Most common challenging in the field of producing these materials is the phase stability. Many studies have tried to solve the obstacles of producing pure ternary carbides and nitrides at low temperatures .Our research results are coincided with the Authors. Lots of variables seem to play an important role. Along the line of element concentrations, some studies have investigated, however, none of them gives the real effect of that issue on the stability of ternary ceramics[Wang,2005].It has been tried in this research to study the effect of Al in the Ti-Al-C system on the phase evolution by limiting the pathway reaction which leads to intermediate phases and changing the temperature starting from low temperature until phase appear at high temperature with changing the Al concentration to understand the direction of the reaction.

Experimental Works:-

At different concentrations, Ti-Al-C have been mixed (10% Al-80%Ti-10%C), (20%Al 70%Ti10%C)and (30%Al-60%Ti-10%C).A mixer is used for 30 minutes to achieve pressing with 30 ton maximum pressure. Cylindrical steel die(made from tool steel)used in compacting process with upper and lower punch. A paraffin was used as a binder in this work at certain powder composition to provide strength to green compact.Furnace used for sintering process atdifferent sintering temperatures (600C°-800C°-1200C°-1400C°) with rate 20 c/minute and is held for three hours, finally the cycle cool down to room temperature inside the furnace sample was held on heat resistant plate inside the furnace.X-ray diffraction tests are done for all samples before and after the sintering practices. Figure (1–8) shows the diffraction patterns obtained for all samples made by concentration distribution and molar ratio distribution. The phases produced as a result of sintering process a reanalyzed by computer analyzer program named International Center for Diffraction Data (ICDD) and by (EXCELL) program in hand way.

Concentration Samples result by using X-ray :

Different concentrations ere (10% Al-80%Ti-10%C),(20%Al-70%Ti10%C)and (30%Al-60%Ti-10%C) subject to a different sintering temperatures (600C°-800C°-1200C°-1400C°). As seen in figure (1), the phase development shows that at 600°C the TiAl and TiAl intermediate phases are presented with amount of non reacted carbon. The Alconcentration has affected the crystallography (peak sharpness) of bothphases. A small amount of non reacted Ti has been found in some samples.

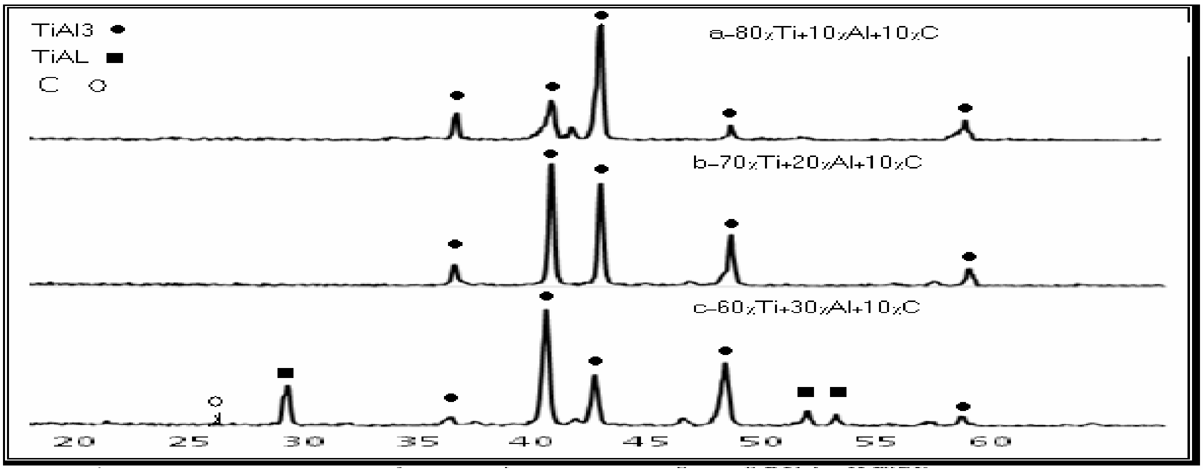


Figure (1):shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at600°C for 3 hrs.

Figure (2) shows the increasing of the sintering temperature $TiAl_3$ which appears to be the dominate phase. The perviskite structure of Ti_3AlC tends to appear in a little amount with the rest of TiAl3.

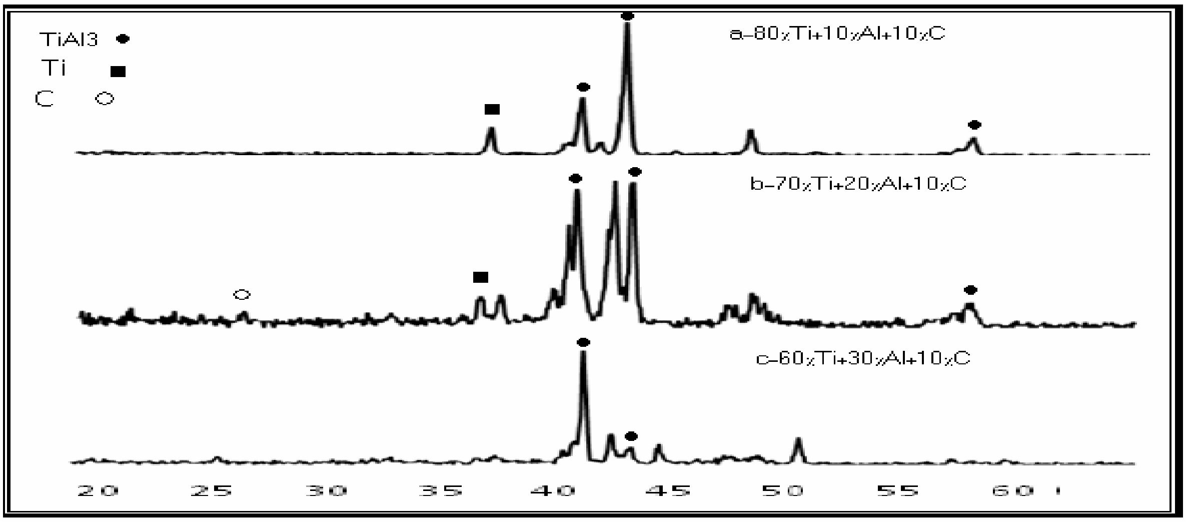


Figure (2):shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at 800°C for 3 hrs.

The following figure (3) shows the structure of TiAlC (perviskite structure) which appear to be more dominated especially with decreasing the Ticoncentration.

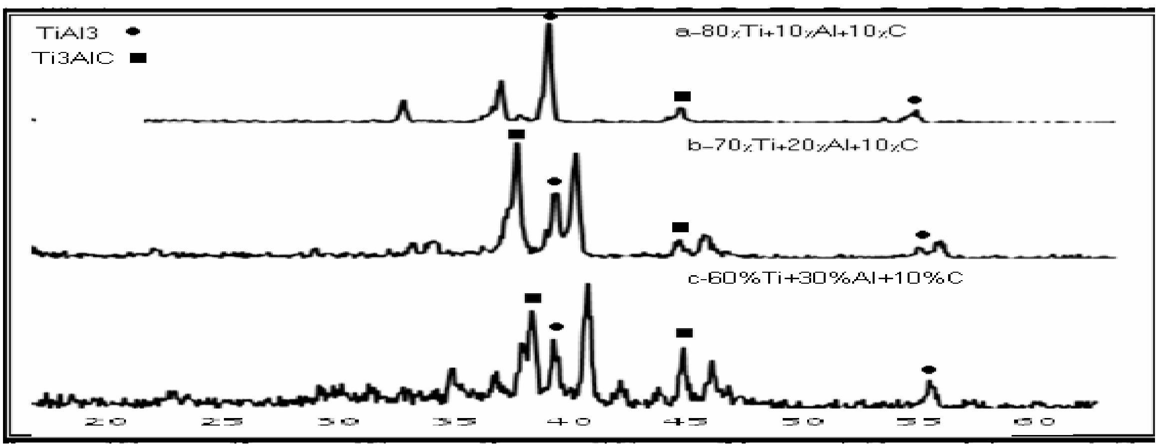


Figure (3):Shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at1200°C for 3 hrs

While, figure (4) shows the appearance of Ti2AlC and TiAlC and some TiC is also presented.

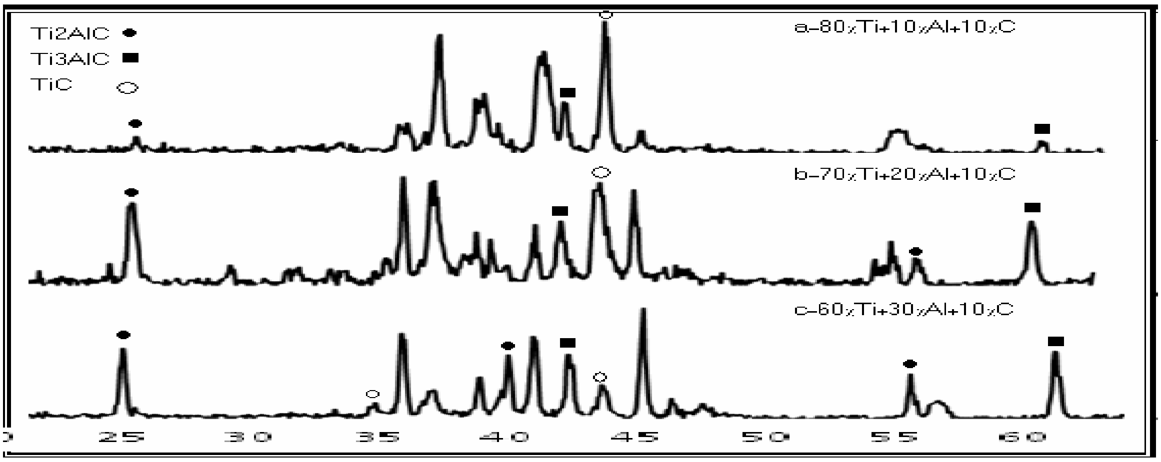


Figure (4):shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at 1400°C for 3 hrs.

X-ray Result for 2:1 Molar Ratio Samples:

The reaction at low temperature shows a group of phases TiAl3 ,TiAl and TiAl which appear to be more dominated, and there is a small amount of pure Ti as in figure (5).

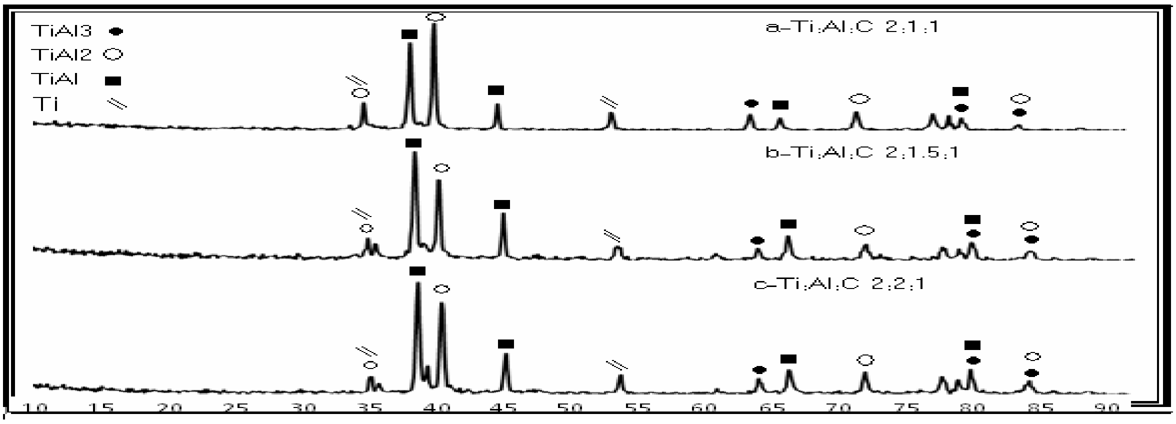


Figure (5):Shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at 600°C for 3 hrs

By increasing the temperature, the crystallization of TiAland TiAlis increase and becomes more dominated, except for TiAl because of low percentage of Al to Ti as in figure (6).

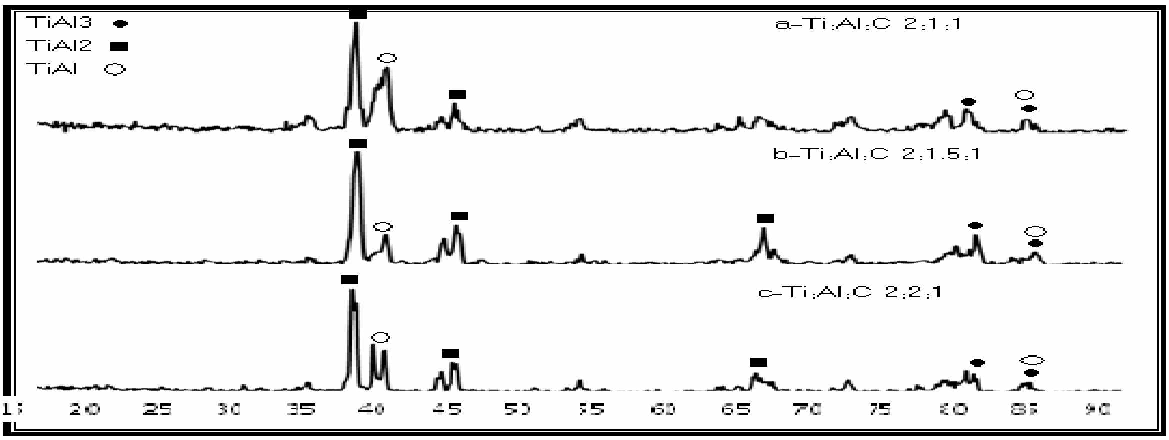


Figure (6):Shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at 800°C for 3 hrs.

While figure (7) shows Ti2AlC phase appears with rest of TiAl3.

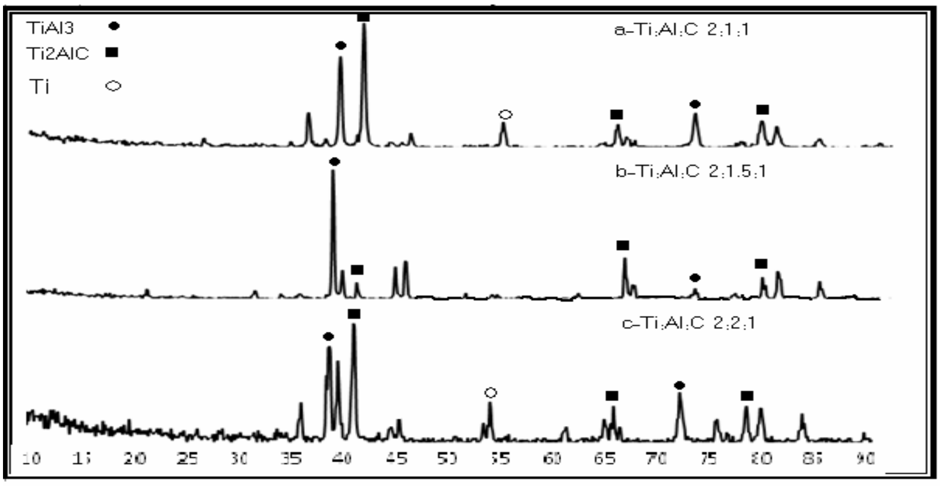


Figure (7):Shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at 1200°C for 3 hrs.

$TiAl_3$ disappears at high temperature and, Ti_2AlC tends to be more dominated with little amount of Ti_3AlC_2 and TiC as in figure (8).

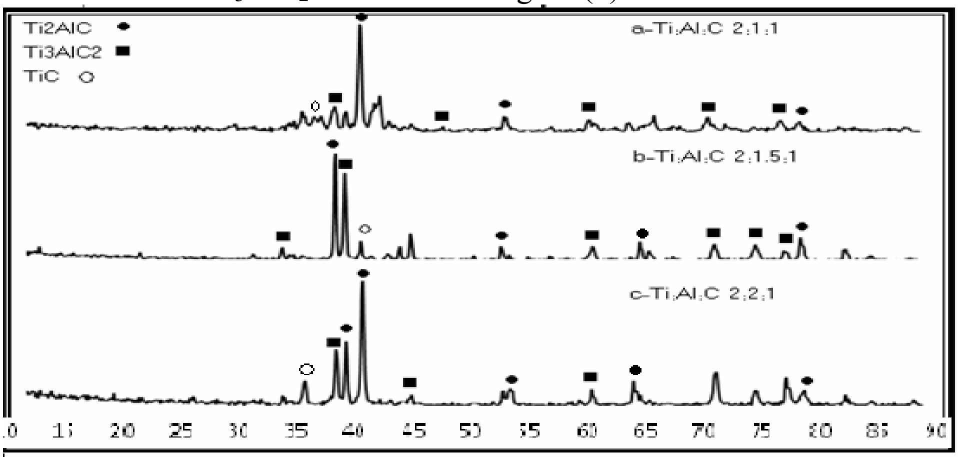
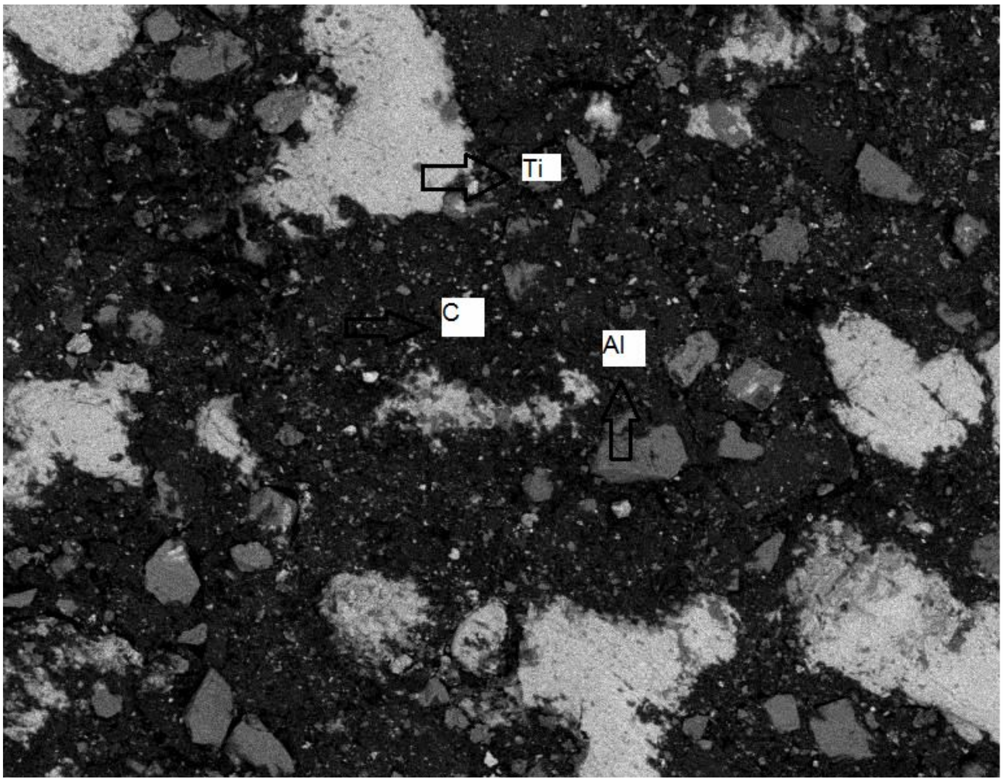


Figure (8):shows, XRD Patterns of Samples Pressed at 30 ton and Sintered at 1400°C for 3 hrs.

It is observed that the SEM micrographs, after the sample are ground by SiC emery paper with different grits starting from 600 and 800 grit , get flat and scratch free surface without coating by carbon. Finally, these samples were polished with smooth cloth as shown in figures (9 -11). The pack scatter shows the different in color which may give an indication about the appearance of most phases[Songlan,2007].



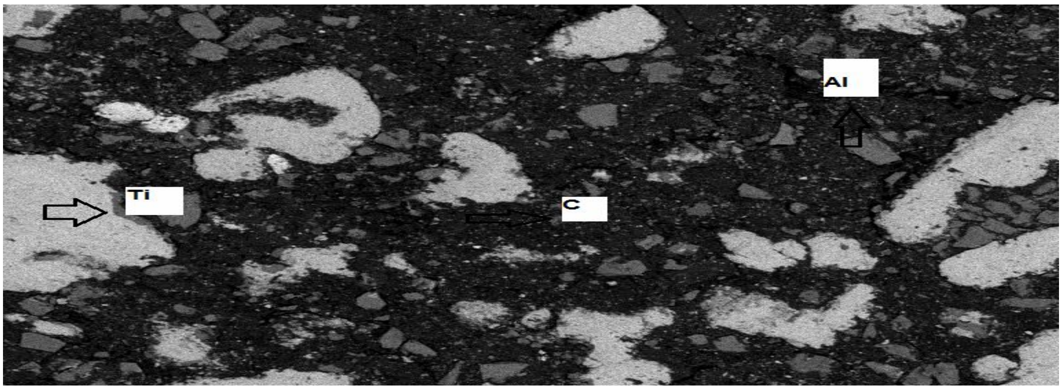


Figure (9):shows, SEM for Ti:Al:C=2:2:1 Hot Compacted at 600°C and Sintered at 1400°C for 3hrs

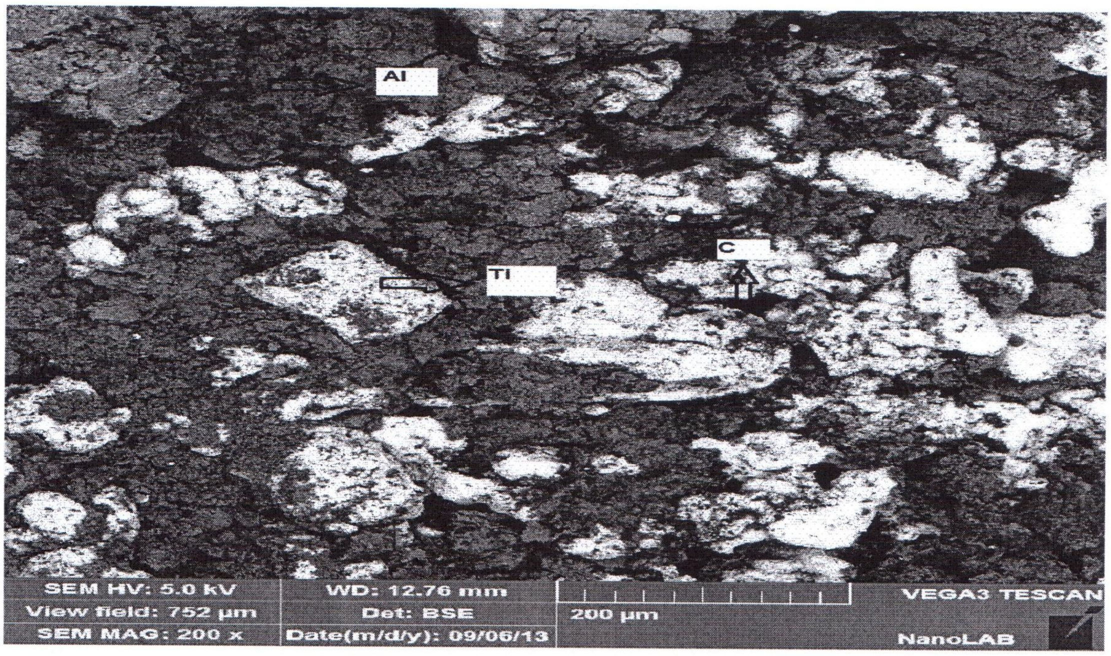
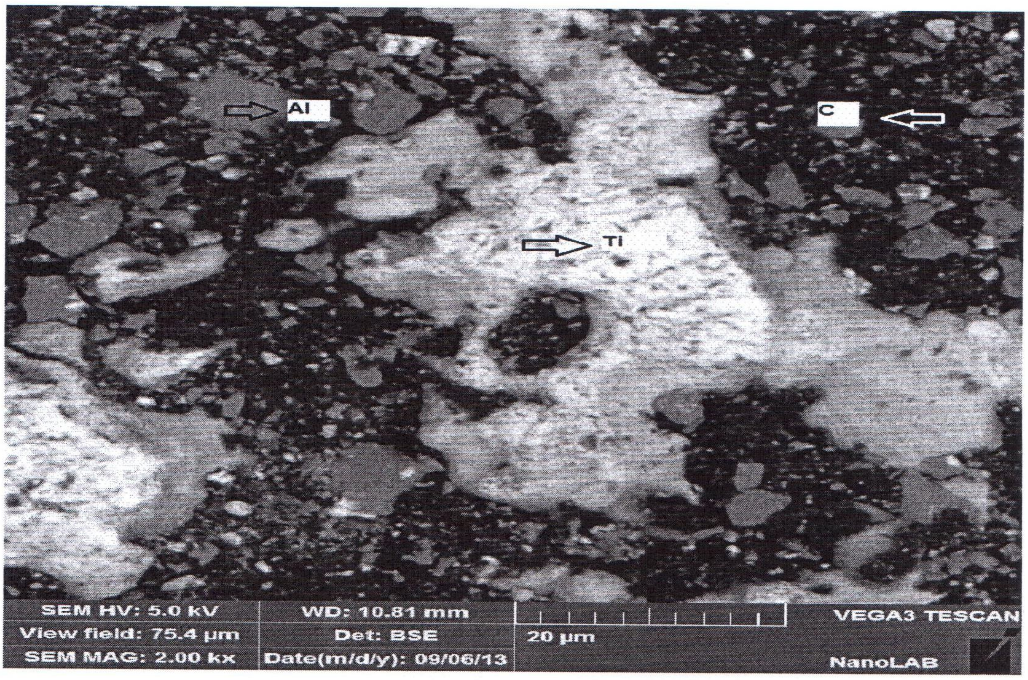


Figure (10):shows, SEM for 60%Ti+30%Al+10%C Cold Compacted and Sintered at 1400°C for 3hrs.



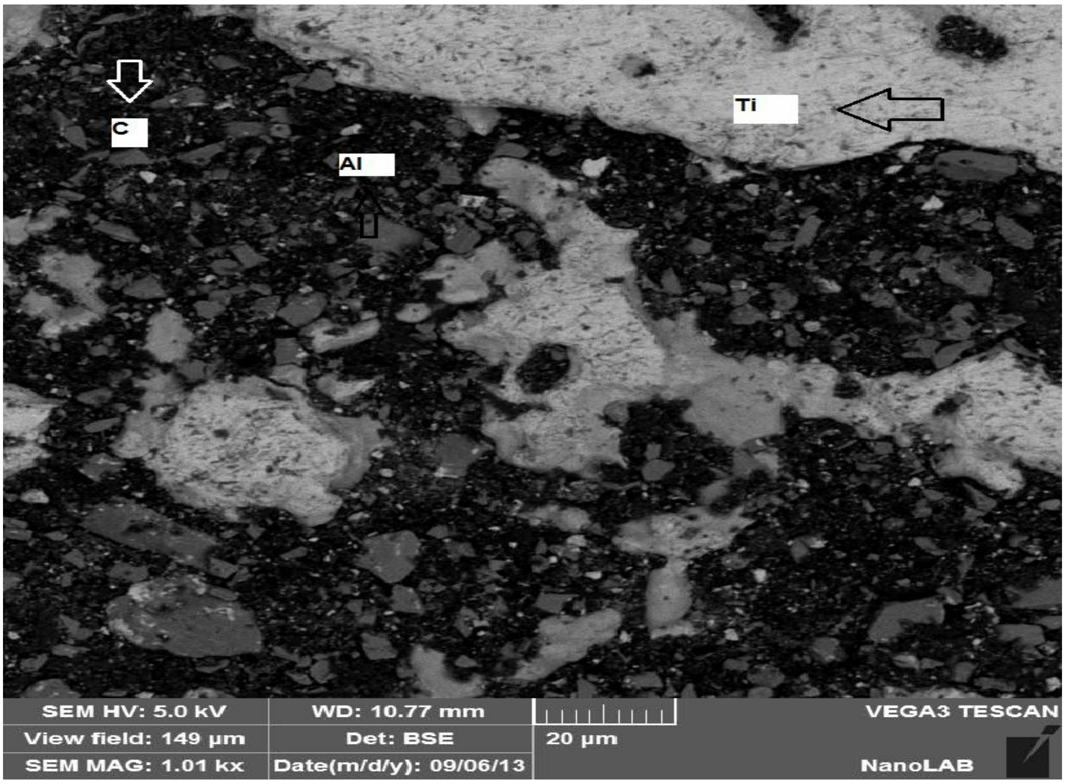


Figure (11):shows, SEM for sample Ti:Al:C 3:2:2 Cold Compacted and Sintered at 1400°C for 3hrs.

Optical Microstructure of different Samples :

Also, the microstructure is studied by an optical microscope in order to see the grains and the phases existing .The optical microscope micrographs is observed in figures (12,13). the magnification are 1450 and the scale of 100µm. The pores are mostly the dark regions [Gauthier *et. al*.,2009]

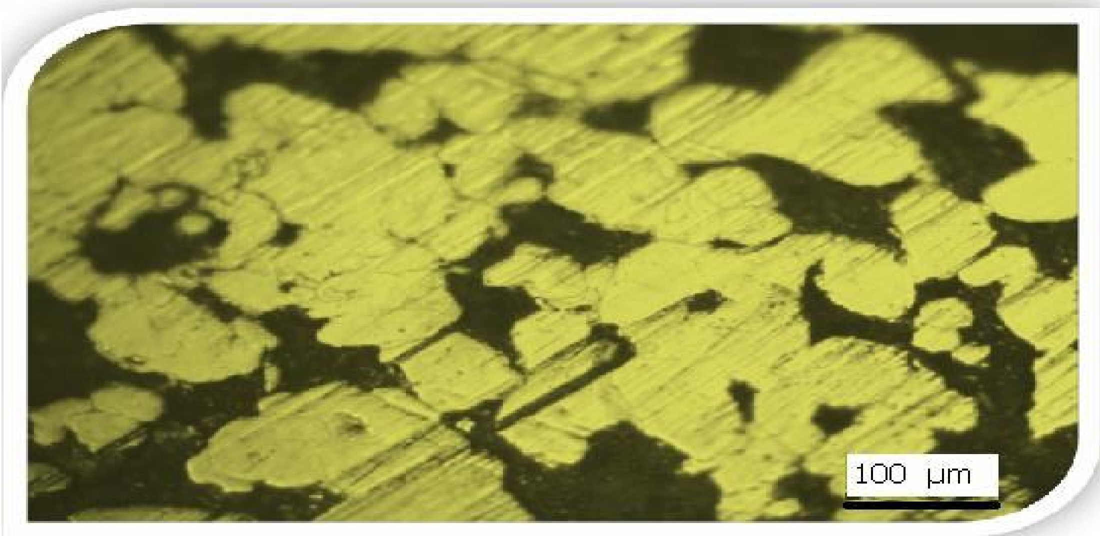


Figure (12):shows, Optical Microstructure of Sample Ti:Al:C 2:2:1 Cold Compacted and Sintered at 1400°C for 3hrs with 145x.

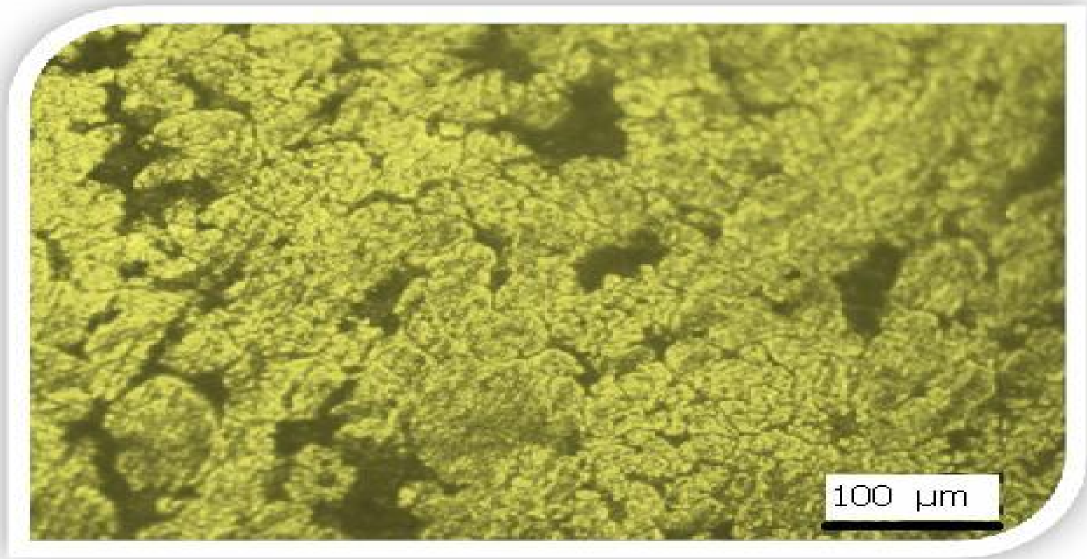


Figure (13):shows, Optical Microstructure of Sample Ti:Al:C 3:2:2 Cold Compacted and Sintered at 1400°C for 3hrs with 145x.

Mechanical Properties

1- Density Test :-

The density percentage is measured by using Archimedes method. This method includes measuring the weight of dry samples, then the samples are immersed in water for 24 hours, then weight the samples after cleaning their surface with cloth, and finally we weight the samples while they are placed in basket and immersed in water. The obtained results are represented, graphically in figures (14,15)which obtained that the density approximately decreased when the temperature was increased .

The density is measured according to this formula:

Density% = [(Ws –Wd)/(Ws –Wn)] *100 ……. (3.1) [Yong *et. al.*,2008]

where: Wd= weight of dry sample. Ws= weight of saturated samples. Wn= weight of immersed
sample.

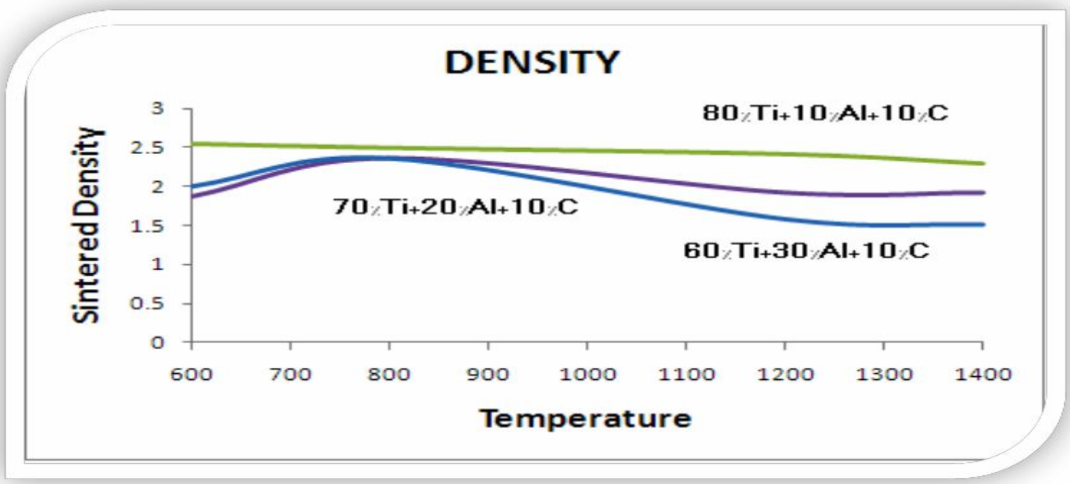


Figure (14):shows, Sintered Density of the Samples with Concentrations Distribution.

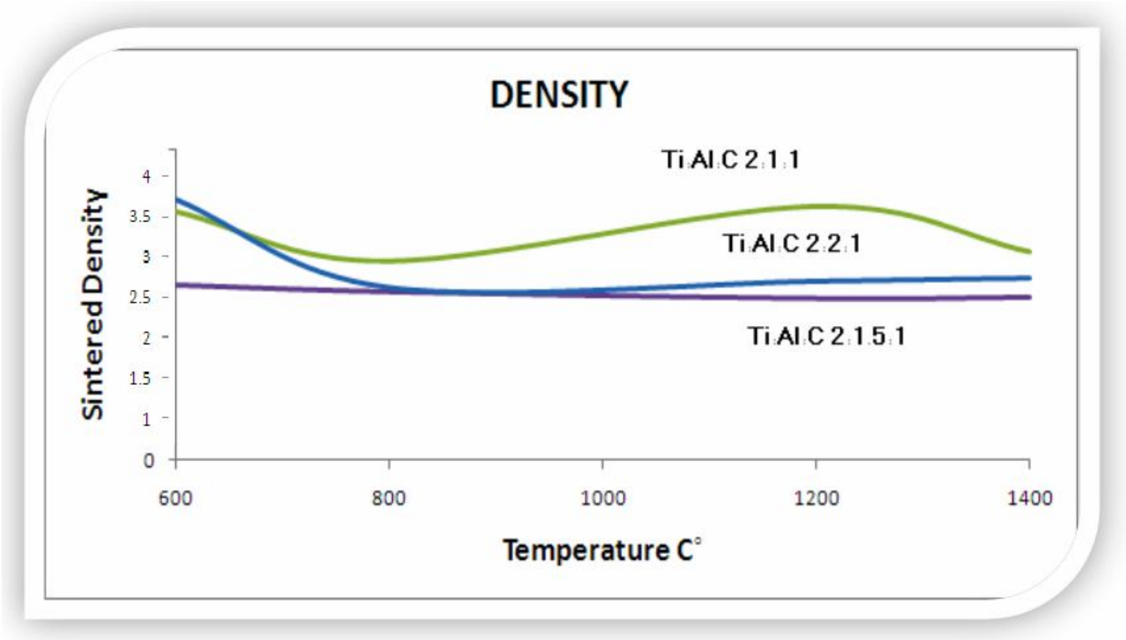


Figure (15):shows, Sintered Density of Samples with Molar Ratio Distribution and Cold compaction.

2- **Hardness Micro Test :-**

For these ceramics, the hardness has been taken by using Vickers micro hardness tester type (TP µP-A, HV-1000) which is located at then corrosion Laboratory[Yong *et. al*.,2008] . The average of 3 readings is taken for samples with diameter of 10mm and 4mm in height . All hardness values are taken at a load of 1Kg. The results obtained are represented graphically infigures (16,17).

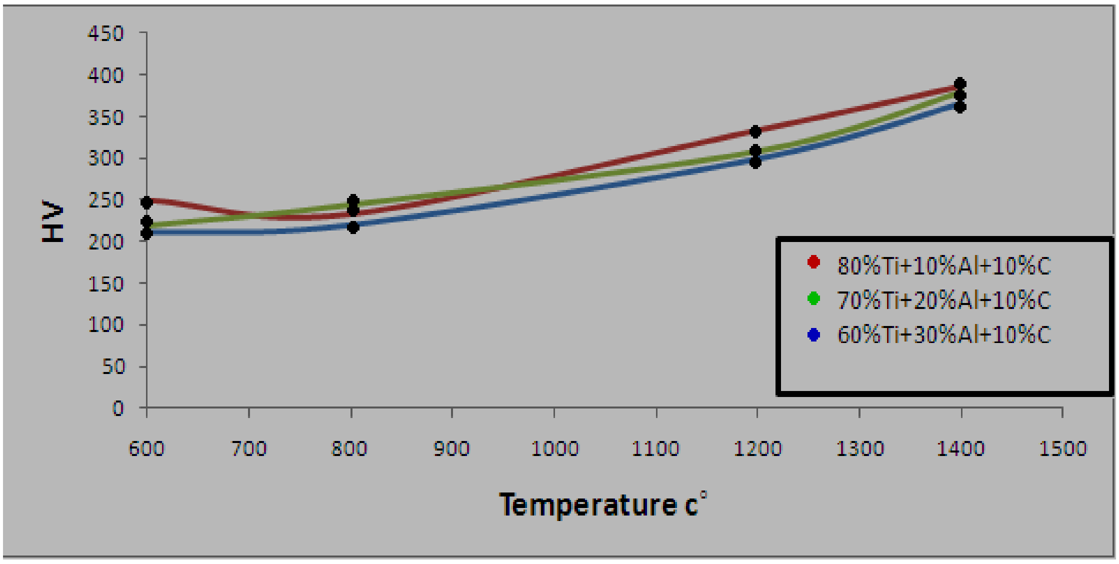


Figure (16): shows, Hardness Value for Samples with Concentration Distributions.

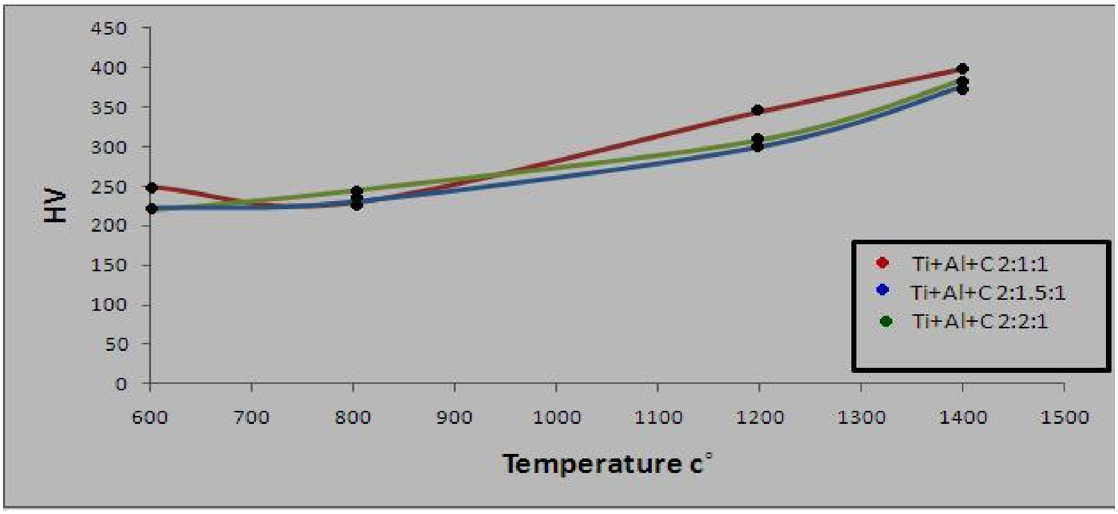


Figure (17):shows, Hardness Value for Samples with Molar Ratio Distribution and Cold Compacted.

At the two systems, the hardness value that cold compacted were closed in the values, it shows increasing when temperature increase due to the phase existence

Conculation Results :-

Microstructure, XRD, SEM and the results show the effect of Al concentration during different temperatures start from 600C°.Other phase appear when the Al concentration increase due to the high Al percentage as shown in figure (1), figure (2). Some of rest of Al melt at 800C° and the TiAl is the dominated phase, by increasing temperature and Al concentration the perviskite structure appears due to sufficient energy and temperature to start the reaction as in figure (3). In figure (4) when the Al concentration increase some of TiC are presented with the other MAX phases, which are a family of ternary carbides and nitrides. The name Mn+1AXn describes the elements included where M is a transition metal, A is mainly a group 13 or 14 element, and X is C or N.The stoichiometry can vary (n = 1, 2, or 3) leading to M2AX, M3AX2 or M4AX3 phases.

The Nanolaminate structure of MAX phase can be classified as a MX blocks alternative with the A metal. Which give the both properties of metals and ceramics, this is caused by the formation of the intermediate phases to final phases. Finally ,the phase evolution is shown by increasing the temperature, the ternary carbides start to be more stable at high temperature instead of intermediate phases that are the most stable at low temperatures.

While, in figures (5) and (6),At 600C°and 800C° there is no clearly effect of Al ratio on the system. This is due to the closely phase appearance in three different molar ratios thus by increasing temperature up to 1200C the intermediate phases transform to final phases due to the activation thermal and kinetic energy, as in figure (7), Finally, by increasing the Almolar ratio, the MAX phase evolution is affected in the direction of decreasing the crystallite of the ternary carbides. the molar ratio distribution shows a clear direction of reaction that leads to the formation of MAX phases.

Finally, one could conclude that the Al concentration may play avery important role in the phase stability in molar ratio systems.

Conclusion :-

The effect of Aluminum was studied on phase evolution and the effect of temperature of Ti-Al-C ternary system, the following conclusions could be drawn:

1- Aluminum had played a very important role on the formation of the intermediate phases and the ternary carbides.

2 -The applications of water resource were improved by the concentration and proved structures, that alloy was especially in corrosion resistance and under water protected structures and proved pipes because the Al created the layer protects this ceramic from hard nature conditions.

3- The molar ratio distribution plays an important role more than the concentration distribution in determining and exacting the finalphase appearance such as in the Ti 2AlC and Ti3AlC products.

4- One reaction direction given leads to the final MAX phase directly without any chances for the formation of another phases such as TiC.

5-The sintering temperature was increased with Al concentration, the MAX phase such as in the Ti3AlC product, showed more domination than the lower Al concentration due to the delaminating of intermediate phases such as TiAl and reacted with the rest of elemental powders to produce the ternary carbides.

6- Also, the value of the density was changed due to the compaction process and Aluminum concentration , the high percentage of Al gave some bonding at low temperature which increase the density value whileat high temperature the uncompleted formation of ternary phases decrease the density and could cause distraction in the system due to the lower time of sintering.

7- At the three systems, the hardness value that cold compacted was closed in the values while in hot compaction system, it showed increasing when temperature increased due to the phase existence.

8- Therefore, the concentration improved the applications water resource structure, that ceramic was especially in corrosion resistance and under water proved structures and proved pipes because the Al created the layer protected this ceramic from hard nature conditions. The result of these characters that, the Al metal led to use this ceramic in loading bearing such as bridges and high impact resistance such as dams.

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