Effect of Ca Addition on the Microstructure and Mechanical Properties of Al-Cu-Mg Alloy

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Received on:22/2/2015 & Accepted on:30/8/2015

Abstract:

In this research the effect of pure (Ca) element addition in different percents of (0.3,0.6,0.9%) on the microstructure and mechanical properties of Al-Cu-Mg alloy were studied .The Alloys were produced using sand casting and heat treated using (T6) treatment which involve [solution treatment, quenching, artificial aging], aging process were carried out at 220° C for different periods of time ranging between (5min to5hr). Also the work involves a study on the effect of (Ca) addition on both grain size as well as the resulting phases before and after heat treatment, samples were examined using optical microscope, Scanning Electron microscope, X-Ray diffractometer and image-J software to estimate the average grain size. The results of hardness and tensile tests for (0.6% Ca) and 4hr aging show the best response as compared with the other alloys. The hardness and strength values have been changed from (78.29HV),(110.57Mpa) respectively during (30min)at 220°C for (non-Ca) content alloy to(125.9HV),(164.2Mpa) for (0.6%C) alloy. X-Ray diffraction , results shows the basic phase that forms is Al2CuMg (S``, S` phase) which is considered as the main strengthening phase in Al alloys, and it was found that (Ca) addition leads to form (Al₄Ca) and (Al₂Ca) which delay the alloy response to precipitation hardening by delaying the formation of (S)phase. Also (Ca) addition in the range of (0.3- 0.6%) gives the refining effect as shown from image-J results While increasing Ca content up 0.9% wt has resulted in a reduction in the grains refining which leads to a decrease in hardness and tensile strength, From these results the decrease in tensile strength with increasing Ca content over 0.9% seems to be attributed to the reduction in elongation caused by the occurrence of micro porosity due to (Ca) addition.

تأثير اضافة الكالسيوم على البنية المجهرية والخواص الميكانيكية لسبيكة مقدمة:

في هذا البحث تم دراسة تأثير اضافة عنصر الكالسيوم بنسب مختلفة (0.3, 0.6, 0.9) على البنية المجهرية والخواص الميكانيكية لسبيكة (ألمنيوم- نحاس مغنيسيوم), حيث تم انتاج هذه السبائك باستخدام السباكة الرملية وعوملت حراريا باستخدام المعاملة الحرارية (T 6) والتي تتضمن (المعاملة المحلولية, الاخماد و التعتيق الصناعي). وتم اجراء عملية التعتيق عند درجة 2200% ولفترات زمنية

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https://doi.org/10.30684/etj.2015.108833

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مختلفة (كدقائق- 5ساعات). تضمنت الدراسة ايضا تأثير اضافة الكالسيوم على كل من الحجم الحبيبي اضافة الى الاطوار الناتجة قبل وبعد المعاملة الحرارية باستخدام المجهر الضوئي , المجهر الالكتروني الماسح , جهاز فحص حيود الاشعة السينية وبرنامج Jimage-J لحساب معدل الحجم الحبيبي. اظهرت نتائج فحص الصلادة والشد ان السبيكة الحاوية على 0.6% Ca تمتلك افضل استجابة ولزمن اطول مقارنة بالسبائك الاخرى , حيث تغيرت قيم الصلادة والمقاومة من HV الم5.7 mpa 110.57 للسبيكة 30 دقيقة عند 220% للسبيكة الخالية من الكالسيوم الى 30% Ca تمتلك افضل استجابة ولزمن اطول الحاوية على 0.6% مع من 2000 وبأستخدام فحص حيود الاشعة السينية اظهرت النتائج ان الطور الاساسي الحاوية على 0.6% Ca من 2000 وبأستخدام فحص حيود الاشعة السينية اظهرت النتائج ان الطور الاساسي الناتج هو الطور (S) ذو التركيب الكيميائي (CuMg2AI) والذي يعتبر احد الاطوار الاساسية المسؤولة عن عملية التقوية في سبائك الالمنيوم. وقد وجد بأن اضافة الكالسيوم ادت الى تأخر الاستجابة الى عملية الاصلاد بالترسيب نتيجة لتكوين المركبات (A14C) , (A14C) والتي تعمل على تأخير تكوين الاطوار الوسطية.

كذلك فان اضافة الكالسيوم بالمدى (0.3-0.6) % ادت الى حصول تنعيم حبيبي كما ظهر من نتائج برنامج (image-J) بينما زيادة نسبة الكالسيوم الى 0.9% ادى الى نقصان في التنعيم وبالتالي ادى الى نقصان في قيم الصلادة ومقاومة الشد وهذا النقصان يمكن ان يعود الى الانخفاض الحاصل في قيم الاستطالة والناتجة من تواجد المسامات المجهرية والناتجة من اضافة الكالسيوم.

INTRODUCTION

luminum is the world's most abundant metal and is the third most common element, comprising 8% of the earth's crust[1]. The versatility of Aluminum **L** makes it the most widely used metal after steel. The most important features of Aluminum are high electrical conductivity and its strength to weight ratio which is superior to steel[2]. Also Aluminum has a high corrosion resistance which is result from the high reactivity of Aluminum with oxygen that lead to the formation of a protective layer to protect the metal for corrosion[3]. Although the previous features Aluminum itself is not hard enough for many technologic and commercial applications, one frequently used method for sorting out this problem consist in alloying other elements, these elements solved into Al matrix are able to diffuse during the artificial aging heat treatment thus leading to the formation of intermetallic phases precipitates that can serves as a barrier for dislocation movement and so improve hardness[4]. Al-alloys have much higher mechanical strength as compared with pure Aluminum which gives draw interest in the structural applications, especially aircraft and cars plants [5]. The principal characteristics and applications of the 2xx.x series of aluminum cast alloys include: heat treatable sand and permanent mold castings; High strength at room and elevated temperatures; some high-toughness alloys; aircraft, automotive applications/engines; representative alloys are: 201.0, 224.0; and approximate ultimate tensile strength ranges : 19 to 65 ksi (130 to 450 MPa) [6]. Calcium has a very low solubility in Aluminum, the solid solubility of (Ca) in (Al) was given as about 0.6% wt at 616°C and about 0.3% wt at room temperature [7]. An interesting group of alloys containing about (5%Ca and 5%Zn) have superplastic properties .Calcium and Aluminum are used by the metallurgical industry for a variety of purposes [8]. In many binary systems containing alkaline earth elements stoichiometric intermetallic compounds often form with melting temperatures much higher than those of the constitutive pure elements.(Al-Ca) system is one of these with a very high melting temperature compound (Al2Ca) as compared with those of pure (Al) and (Ca). (Al2Ca) exists in the molecular like and it is called (associates) [9].

Many researches have been done to improve the mechanical properties of (Al) alloy by adding different kinds of the alloying elements. In 2001 the researcher (D.H.XIAO) and others[10] studied the effect of Cu element addition on the mechanical properties of (Al-Cu-Mg-Ag) alloy (about 4-8%Cu), and they noticed an increment in the tensile strength with a slight decrement in the elongation The high strength of the alloy containing 8%Cu results from the high density of θ' and Ω precipitates .Also Cu increases the stability of Ω phase but reduces its volume due to the formation of the secondary phase precipitates of θ that leads to alloy strengthening.

In 2003the researcher (K. Raviprasad) and others [11] studied the effect of (Ag, Si) addition on the precipitation process in an (Al-2.5Cu-1.5Mg), they have demonstrated that Ag ,Si addition produces multi-component clustering reactions. Also they found that the alloy with Si addition or Ag addition only as compared with that which contains both Ag+Si has much lower hardness than the other one because of the rapid hardening process in the presence of Ag and Si.

Also In 2006 the researcher (Z.Chen) and others[12] studied the effect of 0. 2wt% addition of (Sc) on the ageing behavior to a base Al-2.5Cu-1.5Mg and they found that the peak hardness increases by ~1.2%. and the addition of (Sc) has also refine the dispersion of GPB zones and the simulation results reveal a strong tendency for Sc to cluster with vacancies and Mg atoms.

In 2009 the researcher (N.Afify) and others [13] studied the precipitation kinetics in supersaturated Al-2%Cu-1% Mg alloy .The fine –scale precipitates that occur during aging have been investigated during natural aging at room temperature and it was found that during the aging treatment the super saturation is gradually reduced. The strength increases when fine coherent and/or semi coherent precipitates nucleate. The kinetics of the early stage precipitates were found to be controlled by the migration of Cu and Mg atoms in the matrix. Whereas, the later precipitates as S``, S `and S are controlled by diffusion of Mg and Cu in the alloy. Also in 2012 the researcher(. H. N Girisha) and others[14] studied the effect of magnesium on strength and microstructure of Al-Cu-Mg alloy The micro-structural analysis result shows that 2 % addition of Mg reduces the 20% grain size and 21.52% dendrite structure. The tensile strength and hardness increase with % of Mg. The addition of 2% Mg increases tensile strength 57.9% and hardness of 25%. Aging specimens showed that 1% of Mg has more influence on grain refinement and mechanical properties due to smaller the grain size.

Experimantal Work

Al-2.5%Cu-1.5% Mg alloy with different Ca content were prepared by sand casting using the pure elements by weighting wires of 96.7% pure aluminum of about 340g and by the corresponding quantity of its alloying elements for weight ratios required for each alloy separately, according to the proportion of calcium added the weights where as follows: Copper: 8.5 g, magnesium: 5 g, the weights of the added calcium were respectively (1.25, 2.25, 3.5)g. A gas furnace was used to melt the alloys at 750-800C, the melt was cast using a sand mold. Homogenous annealing was carried out on the alloys at 413°C for 1hr in order to ensure a homogenous distribution of the alloying elements and grain size as much as possible to prevent the dendrite structure and to improve their machine ability [15]. Solution treatment was carried out at 500°C for 1hr in an electrical resistance furnace, then the alloys were rapidly quenched in cold water to form a super saturated solid solution of α and

Artificial aging was performed using an electrical resistance furnace at 220 °C for different periods of time, [5min,30min,1hr,3hr,4hr,5hr].After this process the samples were prepared for hardness test to determine the optimum hardness(peak hardness) values and the response of the alloys to this treatment. Optical microscope (EP-Type2) was used for the micro structural studies of the as cast and heat treated alloys. The specimens were grinded using grinding papers of different sizes(180,220,320,500,1000) polished with polishing(diamond and lubricant) agents and etched with (0.5% HF +99.5% distal water) for metallographic analysis. Hardness test were carried on using Vickers micro hardness testing machine and the tensile test were carried out using WDW-E200 model universal testing machine. The chemical composition of the alloys tested using X-Ray fluorescent (XRF) device and the results are shown in Table (1).

Results and Discussions:

1- Microstructure examination and mechanical properties study for the as cast alloys:

Figure 1 shows the microstructures of the alloys produced after pouring process, in these micrographs it's possible to observe (Cu) and (Mg) precipitations in Al-rich light regions. as well as the intermediate phases as a continuous net which surrounds the grains and these precipitates in a cordoning to X-Ray diffraction results as shown in figure (11) are (S) phase of the chemical composition (Al2CuMg) which are not soluble during casting process. Also it can be noticed that the intermediate phases net that surrounds the grains became thicker with increasing (Ca) content up to 0.6% and this may be related to the formation of the new compounds(Al₂Ca,Al₄Ca) which result from of (Ca) addition. From the micrographs in Figure(1) and from the average grain size calculations in Table (2) we can notice that the structure becomes finer with increasing (Ca) content up to 0.6% wt, while reaching 0.9% Ca leads to an increase in the grain size causing a decrease in both hardness and strength. This grain size increment can be attributed by the micro porosity that increases with increasing (Ca) content. Table 2 shows the results of hardness, yield and tensile strength elongations values for the as cast alloys. From this table and from figures (1 a, b, c and d) it is found that the peak hardness at 0.6%Ca reaches 59 HV while it didn't exceed 49.3HV before (Ca) addition this increase is due to (Ca) solubility in Al matrix that ranges between (0.3-0.6%) in addition to its refining effect to the grains because it acts as a nucleation sites to produce a fine grains so the alloy became stronger, But adding (Ca) in excess of its solubility limit in Al (more than 0.6%) can give a harmful effect to the alloy because the addition of (Ca) itself is a especially problematical, because (Ca) is highly reactive with Hydrogen and atmospheric moisture turning to a hydrated form within a short period of time leading to produce the microporosity due to the hydrogen pick up[18], in addition the increment in the grain size by adding 0.9%Ca all weaken the mechanical properties. Also from table (2) and it was found that both the yield and ultimate tensile stresses increase with increasing (Ca) content to 0.6% and reach a maximum value of (78.85 Mpa) and (90Mpa) respectively for A3, while it doesn't exceed (65.85Mpa) and (74.04Mpa) for A1 without (Ca) addition. This mean that (Ca) element addition up to 0.6% increases the properties while increasing its content up to 0.9% reduce them.

2- Microstructure examination and mechanical properties study for the heat treated alloys:

After homogenization treatment which was carried for residual stresses removal and microstructure homogenization . solution treatment were carried out at 500° C for 1hr, at this temperature all or most of the surplus intermetallic phases are dissolved in Al .This step is followed by quenching in cold water, then aging process is done at 220° C for different periods of time [5min, 30min, 1hr, 3hr, 4 hr, 5hr]. The microstructures for the heat treated alloys are shown in Figures (2,3, 4 and 5). From these figures we can see the dissolution of the grain boundaries in the form of fine fully dispersed precipitates in Al matrix without the presence of grain boundaries clearly defined .While aging process proceed these particles become thicker and denser due to over aging and finally the grain boundaries reformed in the case of very long aging time after the peak conditions. Also it can be observed that the progressive decrease in the grain boundaries, is a consequence of increasing aging time and it's possible to observe the phase change that occurs when aging time increases. This result clearly indicates that the heat treatment conditions followed in these samples facilitate the diffusion of (Cu, Mg) from the grain boundaries into the matrix and the formation of some intermetallic compounds such as (S``, S`)phases, (CaAl₄ and Al₂Ca) as shown from XRD results figure 12, thus improving the homogeneity of the composition of the phases in the alloys. The microstructure examination of the heat treated alloys figures (6-a) (7-a), (8-a) and (9-a)shows a fine particles of S^{*} or S^{*} Phase(Al2CuMg) which precipitated at a short time (30min) for A1 and for longer time 3hr for A2, 4hr for A3, and 5hr for A4. Figures (6-b),(7-b), (8-b) and(9-b) show the microstructures of the alloys at over aging in which it can be observed that the precipitates becomes thicker and denser than the precipitates at the ultimate hardness value, and this precipitates appear as an incoherent and stable precipitates of (Al_2CuMg) (S) which reduce hardness due to the probability of the dislocations to overcome the precipitates so the alloy will be over aged.

From table (3) The variation in yield strength (0.2%proof strength) of the alloys under aging conditions, we can see that the yield strength of the alloys increases to a peak value at an aging time of (4hr) for A3. Aging for a time longer than 4hr was found to lower the yield strength of the alloy. In the base alloy (A1-2.5Cu-1.5Mg) the variation in yield strength with aging time is not very high . when A3 alloys aged for(5min - 4hr) the yield strength increases with increasing aging time but over this amount a decrement in yield strength was observed. Farther aging beyond the peak – age conditions , the yield strength of the alloy decreased due to overage , the same explanation can be applied for the other alloys with increasing aging time. Where, Ca-containing alloys experience extra strengthening due to age hardening effect and the maximum in tensile strength value is achieved by aging 4hr for A3 at 220 °C. Its further observed that increasing aging time up to 30min for A1, 3hr for A2 .4hr for A3 and 5hr for A4 increases the tensile strength of the alloys . increasing aging time over the previous amounts cause a drop in the tensile strength values under aging conditions.

3- Studying the alloys response to precipitation hardening :

It's an important thing to be in consideration that 2xx.x phase transformation do not occur unless a specific circumstances are satisfied, where temperature and time play the main role in it. Vickers hardness tester were used to observe the response of the alloys to precipitation hardening process as well as tensile test at specific points as

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peak hardness (the ultimate hardness value reached during precipitation hardening). Table (3) shows the results of hardness and tensile test for the alloys after aging and at peak hardness. From this table we can see that (Ca) has a significant effect on the precipitation process, it has a great effect on the peak hardness during T6 treatment where (Ca) addition to Al-Cu-Mg alloys leads to delay the formation of the intermediate phases because of the formation of(Al₄Ca)and (Al₂Ca) as shown in XRD results , which reduces the diffusion rate of Cu and Mg atoms in Al matrix. This delaying leads to the need for a longer aging time to form (S[`]) phase of the chemical composition of (Al₂CuMg) which is responsible for the alloys strengthening thus delaying over aging process that weaken the mechanical properties of the alloys as a result of the formation of (S) phase with a larger size as shown in Figure(6-b),(7b),(8-b) and (9-b) previously, leading to alloys brittleness and therefore weakening its mechanical properties. From table 3 we can notice that the maximum hardness that reached after artificial aging for (A1) was (78.29HV) at 220°C for (30Min), while aging the alloys containing (Ca) element the peak hardness values were low at (30Min) as compared with (A1) which is free from Ca, but its back to be increased after (3hr) for (A2) with (0.3%Ca) to (98.7HV), after (4hr) for (A3) with (0.6%Ca) to(125.9HV) and (5hr) for (A4) with (0.9%Ca) to (118HV) .The maximum hardness value was (125.9HV) for (4hr) at (0.6%Ca), from this it can be concluded that this ratio of (Ca) lead to an increment in hardness about(60.8%) and this in turn lead to increasing the time required to get the maximum hardness value about (Three and a half hours), because (Ca) delay the formation of the strengthening phases (S`), (S``)(Al2CuMg) at a time less than in the other alloys with higher (Ca) content as a result of the formation of (Al_2Ca) and (Al_4Ca) compounds. It can be thought that the peak hardness value is a result of Cu and Mg atoms displacement inside the crystal structure of (α) solid solution for a small distance, thus forming a two dimensional plate or disk like gathering called (GPB1) which is distributed uniformly in each particle, and this agreed with the suggestion of the researchers Bagaryatsky and Silcock[16] who consider that the GPB zones first precipitate out from a super saturated solid solution at the early stage of aging. Also soaking the alloys at the aging temperature(at 220 °C) for longer time, leads to the formation of a larger and thicker GPB zones called (GPB-2/S``) with a fine, uniform and coherent structure different from (α). As aging process proceeds, a fine semi-coherent and dispersant particles of S' in GPB2 zones are formed with a chemical composition of (Al₂CuMg) which impedes the dislocation movement leading to an increment in the mechanical properties. Finally these precipitates converted into an stable incoherent compound named (S) (Al₂CuMg), this in turn is agreed with the precipitation sequence according to the researcher Silcock which described as follow[20]:

Supersaturated $\alpha \rightarrow \text{GPB}$ zones (rods) (S ``) \rightarrow S (lath).---(1)[17]

Several explanations for the first stage of hardening were suggested according to the studies on Al-Cu-Mg based alloys with and without micro alloying additions or impurities and with a range of Cu:Mg ratios. Four main mechanisms were suggested for Al-Cu-Mg alloys strengthening which suggest that the strengthening can be occur due to:

1-GP (Guinier-Preston) or GPB (Guinier-Preston-Bagaryatski) zone formation,[17] 2-co-cluster formation,[11]

3-locking of existing dislocations due to a solute-dislocation interaction[18], and

4- The formation of a S" and S' phase.[19]

From the aging curve for the four alloys figure (10) we can suggest that it has two distinct stages: the first stage is a rapid hardness increase which occurs within a short aging time(5min) at 220°C. According to the previous studies it is concluded that the hardness increase during this period accounts for approximately 60% of the total hardness increase observed at the peak hardness condition. After this rapid reaction, the hardness curve exhibits a long plateau, which is followed by a second stage of hardening towards the peak hardness.

4- Effect of Ca addition on the Grain Size :

With the addition of (Ca) to Al-2.5Cu-1.5Mg alloy up to 0.6%wt ,the grains were refined about 28% as shown in table (2) also after aging treatment and at the peak hardness value the grains refinement ratio was 29.5% as shown from table (3) The explanations of this situation can be summarized as follows: The major existing forms of(Ca) in (A)l alloys are (Al2Ca) and (Al4Ca) , the particles of these compounds acts as a heterogeneous nucleation sites of the alloy grains thus the nucleation efficiency is improved and the grains are refined. The addition of(Ca) over its solid solubility in Al over(0.6%wt) causes a decrease in grains refining and this can be related to the increment of the microporosity formation with increasing (Ca) content which reduces the mechanical properties of the alloy.

Conclusions :

From (Ca) addition in different ratios to (Al-2.5Cu-1.5Mg) and after heat treatments the following conclusions are reached :

1- The addition of Ca up to 0.6% wt gives an increment in the yield strength, tensile strength and hardness about (64%), (48%) and (60.8%) respectively. This increment is resulted from Ca effect on the grain refining as well as the fine insoluble compounds that Ca addition produced which strengthen the alloy. While increasing Ca content to 0.9% wt caused a decrease in the mechanical properties of the alloy.

2- XRD results show that the main strengthening phase was (S) of the chemical composition (Al_2CuMg) , as well as (Al_2Ca) and (Al_4Ca) which results from Ca addition.

3- Ca addition up to 0.6% refines the grains about 28% before heat treatment and about 29.5% after aging.

4- Ca addition causes a delay in the alloys response to the precipitation hardening during artificial aging ,because it works as an obstacle to Cu and Mg atoms diffusion in Al matrix by forming AL4Ca and Al2Ca compounds ,therefore the alloys will need a longer time and high temperature to form the strengthening phases.

5- The maximum hardness value(125.9HV) has been reached after 4hr aging for the alloy with 0.6%wt Ca (A3), while the maximum value for A1(without Ca) was (78.29HV) after 30min and the hardness for A2 and A4 were (98.7HV) and (118HV) respectively.

Alloy symbol	Al%	Cu%	Mg%	Ca%
A1	96.11	2.25	1.63	0.006
A2	95.5	2.63	1.66	0.286
A3	95.38	2.47	1.57	0.574
A4	95.3	2.46	1.37	0.889

Table (1) Chemical composition of the Alloys

Alloy symbol	HV (Kg/mm ²)	Ultimate Tensile stress (Mpa)	Yield stress (Mpa)	Elongat ion %	Average grain size (pixel ²)
A1	49.3	74.04	65.85	0.85	75
A2	57	89.75	71.60	0.88	71
A3	59	90	76.84	0.94	54
A4	56	85.3	68.3	0.78	72

Table (2)) Mechanical	properties	of as	cast alloys
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Figure (1) The microstructure of the cast alloys

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Figure(2) A1 microstructures during aging process



Figure(3) A2 microstructures during aging process



Figure(4) A3 microstructures during aging process



Figure(5) A4 microstructures during aging process



Figure (6-a) the microstructure of A1 at peak hardness ,we can see a fully dispersed precipitates in the matrix without the presence of the grain boundaries



Figure(6-b) the microstructure of A1 at over aging time ,the precipitates became thicker and denser

Figure (6) The microstructure of A1(Al-2.5Cu-1.5Mg) after aging



Figure (7-a) the microstructure of A2 at peak hardness, fully dispersed precipitates in the matrix without the presense of the grain boundaries



Figure(7-b) the microstructure of A2 at over aging time, the precipitates became thicker and denser

Figure(7) The microstructure of A2(Al-2.5Cu-1.5Mg-0.3%Ca) after aging

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Figure (8-a) the microstructure of A3 at peak hardness, fully dispersed precipitates in the matrix without the presense of the grain boundaries



Figure(8-b) the microstructure of A3 at over aging time





Figure (9-a) the microstructure of A4 at peak hardness, fully dispersed precipitates in the matrix without the presense of the grain boundaries



Figure(9-b) the microstructure of A4 at over aging time

Figure (9) The microstructure of A4(Al-2.5Cu-1.5Mg-0.9%Ca) after aging

 Table (3) The results of hardness, yield and ultimate tensile strengths ,elongation and average grain size for the alloys at peak hardness values

Alloys	HV(Kg/mm ²)	Ultimate tensile	Yield stress(Mna)	Elongation%	Average grain	Time at
		stress(Mpa)	stress(tripa)		per(pixel ^{^2})	hardness
A1	78.29	110.57	70.68	0.86	112	30min
A2	98.7	125.6	84.56	0.89	98	3hr
A3	125.9	164.2	116.23	1.01	86	4hr
A4	118	139.6	89.1	0.82	94	5hr



Figure(10) aging curve of Al-2.5Cu-1.5Mg-(0.3,0.6,0.9wt% Ca) alloys at 220°C show the variation in hardness with aging time

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Figure (11) XRD results for as cast alloys



Figure (12) XRD results for heat treated alloys

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