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Effect of Zn % and Thermomechanical Treatments on the Mechanical Properties of Al-Zn-Mg Alloys

Dr. Eng. Ali H. Ataiwi*

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Abstract

The aging kinetics and the coupling between plastic deformation and subsequent aging on a set of Al-Zn-Mg alloys having different Zn-content (6.33%, 7%, 7.5% and 8%) are investigated. These alloys are produced with an extreme care during melting, alloying and sampling where the best conditions of alloying are adopted .The response to multi thermomechanical treatments practices are reported, where the combination between rolling practice and aging is monitored by hardness measurements, tensile, impact toughness measurements in addition to x-ray diffraction testing. The study proves that, the zinc percent has a very important role in the precipitation hardening sequence of tested alloy; this effect is concentrated on the speeding up the kinetics of hardening by increasing the density and stability of GP zones and µ'-phase.

تأثير نسب الخارصين والمعاملات الثرموديناميكية على الخواص الميكانيكية لسبائك ألمنيوم_خار صين_مغنسيوم

الخلاصة

تم دراسة آليات التعتيق والموالفة بين التشكيل اللدن والتعتيق اللاحق علـى مجموعـة مـن السبائك الثلاثية ذات السلسلة7000 (ألمنيوم – خار صين – مغنيسيوم) ذات قيم متغيره مـن الخارصين 6.33% , 7% , 7.5% ,8%). حيث تم إنتاج هذه السبائك خلال الصهر والتشبيك وتصنيع العينات بدقة عالية وتم اختيار أفضلها ، حيث تم تسـجيل اسـتجابة هـذه السـبائك للمعاملات الثرموميكانيكية من حيث إجراء الدرفلة و التعتيق و من ثـم قيـاس الصـلادة ومقاومة الشد والصدمة إضافة لفحوصات حيود الأشعة السينية . وقد أثبتت الدراسة أن لنسبة الخارصين دور كبير على عملية التصليد بالترسيب حيث تركز هذا التأثير على تسـريع آليـة التصليد من خلال زيادة كثافة واستقرار مجالات كنيه–بريستون والطور´µ.

Introduction

The precipitation from a solid solution and the related hardening of alloys is one of the most investigated areas of physical and mechanical metallurgy, both from the experimental and the theoretical viewpoint [1]. The Al-Zn-Mg alloys family provides some of the most interesting alloys for light-weight structural applications, in particular in the aircraft industry. Their properties are obtained through a complex thermomechanical treatment, which ends with a solution treatment, a quench, a plastic deformation either to relieve quench stresses or to form these alloys and a multi-step aging treatment, with the usual precipitation

* Dept. of Materials Science, University of Technology.

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2412-0758/University of Technology-Iraq, Baghdad, Iraq

sequence[2].

$$ssss \rightarrow GP_{Zones} \rightarrow \mu'_{Metastable} \rightarrow \mu_{Stable(\ MgZn_2)}$$

where (ssss) is the super-saturated solid solution. GP is Gunier – Preston zones. It is important to note that the ($MgZn_2$) or µ seems to nucleate independently, that is the µ' does not directly transform into µ.

The plastic deformation step is of very importance either in its amount or in its sequence. This step aims either to relieve quench-related internal stresses which would alter dimensional stability during later machining of the alloy, or in order to change the shapes i.e. forming the alloy. This plastic deformation step introduces in the material a large density of dislocations which can alter the precipitation sequence in a number of ways, either by heterogeneous precipitation on dislocations or by modification of bulk precipitation [3]

The current work tries to focus on the importance of the following[4]:

1-Preaging practices, i.e. the initial conditions of the set of alloys from each compositions prior to the applications of mechanical deformation.

2- Post aging, i.e. final conditions after the applications of mechanical deformation.

Experimental Procedure

The chemical compositions of the set of material employed in this work are given in Table (I). These sets of alloys are produced with high attention during melting, alloying and sampling. All these samples were casted in the form of 10mm thickness plates.

Samples required for tensile tests are selected under the following different conditions [5]:

1-As cast samples from each set of alloy.

2-Aged for peak hardness conditions in each type of aging.

3-At peak hardness with or without rolling.

This measurement is made parallel to the rolling direction according to *DIN standard No. 3.4365* [6]. All tests were performed under constant speed (5mm/minute). In order to asses the toughness of the produced alloys, impact tests are conducted. The notched bar impact test samples are prepared also at the same conditions mentioned above, according to *DIN 50115* [7]. All samples prepared for this work, were solution treated for (6 hrs.) at (480ºC ± 5ºC) with (5ºC/min.) as a rate of heating and then cold water quenched. One set of samples from each composition was left for up to 1000 hrs in room temperature in order to be naturally aged (this is termed T4-conditions).

Another set of samples from several compositions, i.e. A, B, C and D was artificially aged at (120, 140, 160, 180 and 200ºC). These samples were aged for up to (20 hrs.) at each aging temperature and then air-cooled to the room temperature (this is termed as T6-conditions).

The cold and warm rolling (190 ˚C) processes were performed using a simple workshop-rolling machine with a single stage. This rolling machine is capable of reducing alloy ingots by 3% in each pass. A detailed TMT plan is shown in Table (II).

Vickers hardness measurements were taken for each sample in different aging time-ray diffraction was performed on samples in peak hardness conditions. Samples for x-ray diffraction were prepared by cutting methods under an effective coolant to avoid the problems of over heating.

Results and Discussion

Alloy (A), in the as cast conditions, was chosen as a base line and additional compositions (B, C and D) (see Table I), were selected so that the effects of the Zn level could be examined.

1. Response of Alloys to Aging Treatments

The fast initial increase in the hardness as can be seen in Figure (1) that shows the Vickers hardness values as a function of natural aging time for alloys (A, B, C and D),.revealing that GP zones may be formed at room temperature immediately after quenching. Deshamps et al reveaded these zones come out 10 min after quenching [8]. Owing to the effect of these zones, the hardness of each alloy increases continuously with the time of natural aging (T4).

The second set of samples that was subjected to artificial aging practices (T6), although it is very clear from Figure (2) that there is a slight improvement in hardness during the first 24 hrs, it is decided to keep the interval between quenching after solution treatments and heating to artificial aging as minimum as possible (i.e. few minutes). After aging for 10 minutes, spherical coherent GP zones can be identified in the matrix [9]

2. Pre-aging Practices & its Effects on Subsequent TMT

In general, the aging response of alloys (A, B, C and D) was reported, the peak hardness for the five aging temperature (120, 140, 160, 180 and 200ºC) are shown in Figure (2). It was decided to choose the aging temperature of (160ºC) as a pre aging temperature on the subsequent TMT for the following reasons:

1-Due to the high diffusion, this temperature resulted in more extensive second phase precipitate and hence resulted in higher peak hardness with shorter aging time [10].

2-Coherent and semi-coherent precipitates availability at the peak hardness conditions.

In order to understand the behavior of the alloys during an aging treatment at (160ºC), it is first important to investigate the aging sequence that may happen within this temperature. The thermal evolution of the process of precipitation at this aging temperature can be divided into three stages:

1-Nucleation of GP zone.

2-Growing of the ordered or metastable µ'-phase.

3-Transformation to a stable phases i.e. µ or ($MgZn_2$).

The hardness of Al-Zn-Mg alloys during the period in which GP zone formation controls the hardening, i.e. the enhanced aging as observed in figures (2) is believed to be due to greater GP zone nucleation, resulting in higher volume fraction of zones, in addition to µ'-phase that is semi-coherent phase.

One of the challenges in this subject is the problem of precipitate instability that is initially present in the material, which involves phenomenon such as dissolution, coarsening and transformation from metastable particles to more stable ones. This problem is quite expected, especially if there is a drop or elevation in aging temperature especially if the rate of heating is not constant during aging process.

Now, the aim of the pre-aging practice is to prepare the alloy to the subsequent plastic deformation step and to ensure that an existing of a sufficient, fine distributed of stable and metastable precipitates that must be available in the next stage of TMT.

All the aging practices are performed with heating at a constant rate (5ºC/minute), which is considered as a low heating rate. One can observe that from many literatures [10] if the rate of heating is low, it may decrease the critical radius of GP zone dissolution. That means the life period of GP zones becomes shorter. So, and due to the short life of GP zones, the nucleation of the µ'-phase during aging can take place on these zones resulting in a fine precipitate distribution. At this point, it must be understood that, the slow rate of heating becomes very suitable during the pre aging treatments since it gives a fine distribution of µ' throughout the microstructure.

The x-ray diffraction helps to some extent in monitoring phases that appear especially at the peak hardness conditions where the testing is adopted. The results of such examination or testing assume that a metastable µ'-phase can be appeared as can be seen in table (III).

3. Rolling the alloys at the Peak Hardness Conditions

Rolling practice at these conditions is of two types:

1-Rolling the pre-aged alloys to the peak hardness with (3%) at room temperature i.e. cold rolling.

2-Rolling the pre-aged alloys with (3%) at 190ºC, which is less than the recrystallization temperature, i.e. warm rolling.

The introduction of dislocations by rolling(strain hardening) after aging induces significant differences in the transformation sequence of the treated alloys [11]

4. Response to Post Heat Treatments

Before going through this subject deeply, it must be remembered that the response to this type of treatment depends mainly on the following factors:

1-Amount and temperature of plastic deformation that occurs prior to this treatment.

2-The consequence stage of the plastic deformation, i.e. after the solution treatments or after pre aging treatments.

4.1 ***Post aging Response of Al-Alloys Rolled at Room Temperature after Pre aging to the Peak Hardness (TMT_1)***

Fig.(3) shows variation in Vickers hardness of A, B, C and D alloys after the post aging treatment at 200°C up to 6 hrs. The selection of this postaging treatment temperature comes as a result of following reasons:

1-It is higher than the solvus line of GP zones and much, lower than recrystallization temperature which is around 317°C.

2-More economic in using the heat treatment furnace.

Fig.(4) shows tensile and impact properties of alloys A, B, C and D at peak hardness conditions after postaging process. These figures show some modifications in yield,ulti-mate strength ,ductility and impact resistance. When comparing these alloys. Generally the poor impact toughness properties(figure 4c) may be as a result of the non-homogeneous dislocations that resulted from the rolling practice. These dislocations may be concentrated at the active slip planes that causes the so called a non-homogeneous planar slip phenomenon [12]. One can ask about the role of increasing zinc percentages in this practice and the answer is so obvious in Figs (4a-c). The figures show that, tensile strength and yield strength can be improved as the zinc percentage is increased. The degradation in impact toughness values becomes small as zinc percentage is increased. This important point may lead to understanding that the cutting process of coherent and semi-coherent precipitates that occur in A-alloy, becomes decreased in B, C and D alloys i.e. the resulted non-homogeneous planar slip becomes less as the zinc percentage is increased ,this leads to slight increment in yield and ultimate tensile properties and reduction in elongation and impact resistance properties.

The increasing of coherent and semi-coherent precipitate density because of the zinc addition increment becomes very useful, since, these precipitates become more resistant to the cutting process, which then results in the losses in impact toughness.

4.2 ***Post aging Response of Al-Alloys Rolled at Moderate Temperature after Preaging to the Peak Hardness (TMT_2)***

A temperature of 190°C was chosen as a warm rolling temperature since it is above the solvus line of GP zones [13, 14] and much lower than the recrystallization temperature of alloys. On peak hardness conditions at preaging temperature of 160°C, most of hardening precipitates predominantly coherent and semi-coherent with a little possibility of appearing incoherent precipitate (μ'-phase). The presence of these types of precipitates facilitates the re-dissolution of the fine coherent precipitates i.e. GP2 zones especially when reheated to 190°C in order to warm rolling the alloys.

The homogeneous dislocation density distributions that imparts by the warm rolling practice leads to a very noticeable modifications in mechanical properties as can be seen in the Figs (5) and (6) as bar chart representations. These figures clearly explain the improvement and good response of the alloys A, B, C and D to this type of treatment.

4.3 Postaging Response of Al-Alloys Rolled at Moderate Temperature at the Solution Treated Conditions (TMT$_3$)

It is notable to use of (Al-Zn-Mg) alloys in aerospace applications. After the solution treatment and a fast quench, the material will experience a plastic deformation of many few percent as that used in this work, or sometimes larger in order to relieve quench induced internal stresses or to form these alloys with some degrees of deformations.

This deformation occurs in a highly supersaturated state. It can be seen that, the dynamic precipitation phenomenon may occur even at ambient temperature (i.e. cold working by rolling). After this deformation step, the material goes into several steps of heat treatments (i.e. postaging treatments at different time interval) in order to develop a fine homogeneous distribution of hardening precipitate (μ'). This fine homogeneous precipitation is in competition with the coarser precipitation on dislocations, and it is of very important to understand in which conditions one precipitate family will dominate over the other. Finally, one has to understand the precipitation hardening, which will result from this complicated microstructure competition. Fig.(7), shows the response of A, B, C and D alloys to the post-aging practice, this figure shows clearly the decreasing of hardness of alloys with the post-aging time.

During the postaging, solutes (Zn,Mg,Cu,…etc) diffuse towards dislocations, because of mutual elastic interactions [15,16]. High diffusivity in the dislocations core drives these solutes to the precipitates lying on the dislocations. This leads to:

1-Rapid growth of these precipitates to very large sizes.

2-Development of a precipitate-free zone that surrounds dislocations.

This combined effect results in average in an increased rate of coarsening of the precipitates and in a decrease of yield and ultimate tensile properties with a noticeable improvement in ductility and impact toughness values as can be seen in Figs.(8a and 8c) .

Conclusions

The main conclusions can be summarized as follows:

1-The zinc level has a great influence on the hardening kinetics and a little effect on the maximum hardness, tensile properties and impact strength that can be achieved in Al-Zn-Mg alloys. Its effect is somewhat diminished in naturally aged samples.

2-Cold rolling at the peak hardness conditions has resulted in a non-uniform response to the subsequent post aging treatments.

3-The non-uniform response mentioned above could be avoided by rolling the alloys either at an elevated temperature in the solution treated conditions, or at an elevated temperature in the peak hardness conditions.

4-Warm rolling (190˚C) the alloys at the peak hardness conditions of alloys of different zinc contents gives very good mechanical properties at the subsequent post-aging practice and noticeably improves the impact toughness.

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Table (I)
Average compositions of as cast Al-Zn-Mg alloys with different zinc content

Material Designation	Zn %	Mg%	Cu %	Fe %	Si %	Mn%	Cr %	Ti %	Al%
A	6.33	1.71	2.47	0.68	0.38	0.07	0.21	0.03	Balance
B	7	1.5	2.3	0.6	0.30	0.01	0.25	0.00	Balance
C	7.5	1.4	2.5	0.5	0.4	0.03	0.22	0.01	Balance
D	8	1.6	2.1	0.7	0.3	0.00	0.23	0.01	Balance

Table (II)
A detailed plan that followed in performing a multi practice of thermo mechanical treatments

Alloy designation	Practice No.	Practice identity	Practice details †
A	1	TMT_{1A}	160-9 + CR(3) +200-6
	2	TMT_{2A}	160-9 + WR(3) +200-6
	3	TMT_{3A}	ST+WR(3) +200-6
B	1	TMT_{1B}	160-8.5 + CR(3) +200-6
	2	TMT_{2B}	160-8.5 + WR(3) +200-6
	3	TMT_{3B}	ST+WR(3) +200-6
C	1	TMT_{1C}	160-6 + CR(3) +200-6
	2	TMT_{2C}	160-6 + WR(3) +200-6
	3	TMT_{3C}	ST+WR(3) +200-6
D	1	TMT_{1D}	160-4.5 + CR(3) +200-6
	2	TMT_{2D}	160-4.5 + WR(3) +200-6
	3	TMT_{3D}	ST+WR(3) +200-6

† **Table key**: the first digit is the aging temperature, second digit represents the aging time, CR is the short name of cold rolling, while the WR is the warm rolling, (digit) represents the deformation percentage, fourth digit represents the post aging temperature, and finally the last digit represents the maximum post aging time.ST is solution treatment

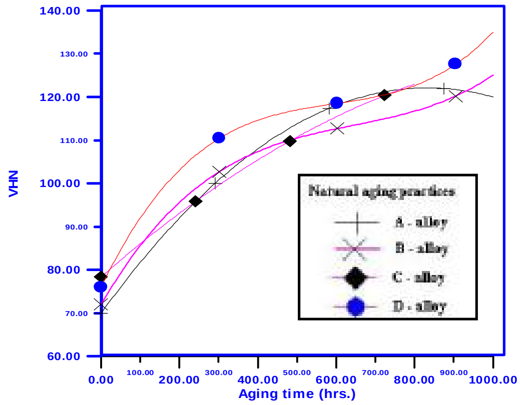
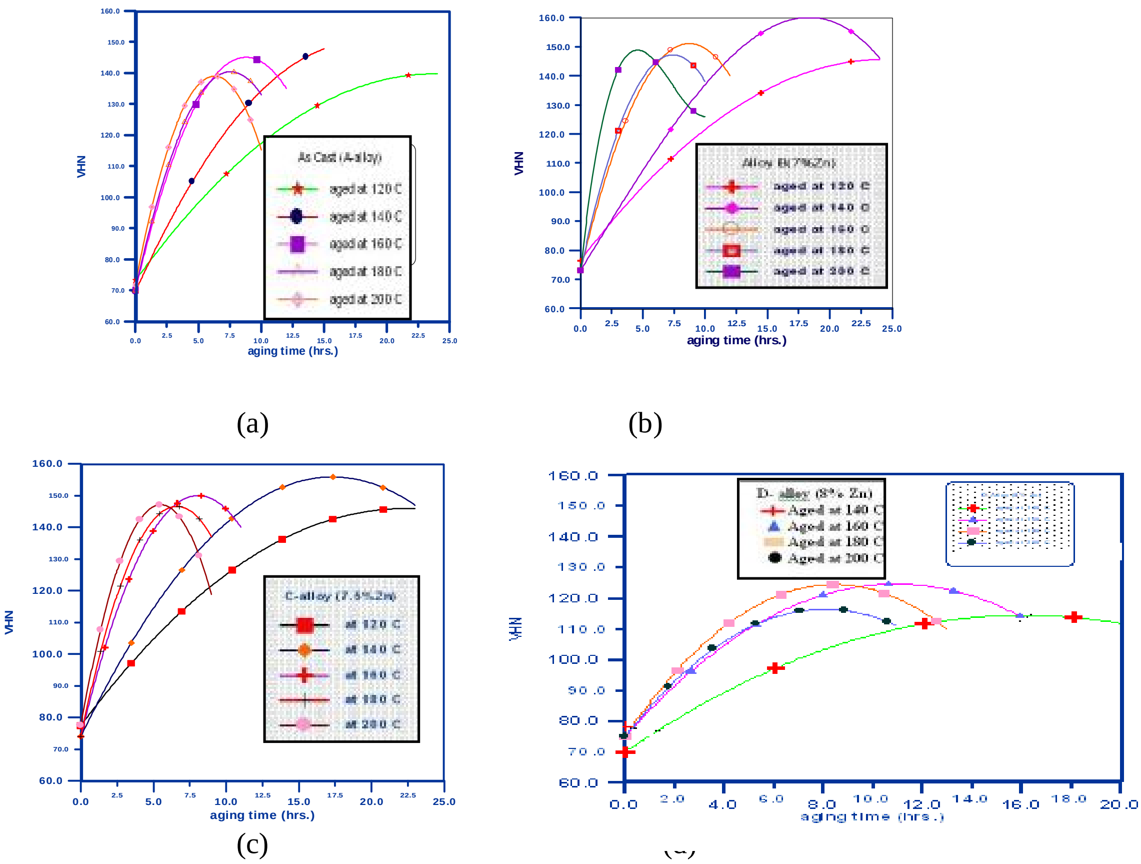


Figure (1) Accumulative diagram shows the response of tested alloys (A, B, C and D) to the natural aging (T4)



(a) (b)

(c)

Figure (2) Relation between Vickers hardness number. and aging time shows the response to T6 at different aging temperatures, of a) A alloy , b) B alloy , c) C alloy , d) D alloy .

Table (III) Sample of X-ray diffraction examination shows the appearing phases in tested B-alloy that subjected to T6 at 160

2θ (degree)	Phase
37.5	$Mg_{32}(AlZn)_{49}$
38.2	Al
41	$MnAl_6$
45.8	$Al_{0.71}Zn_{0.29}$
83.85	$Al_{0.71}Zn_{0.29}$
88.2	Al_5Fe_2

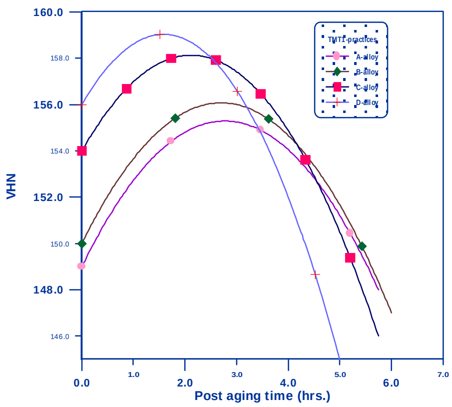
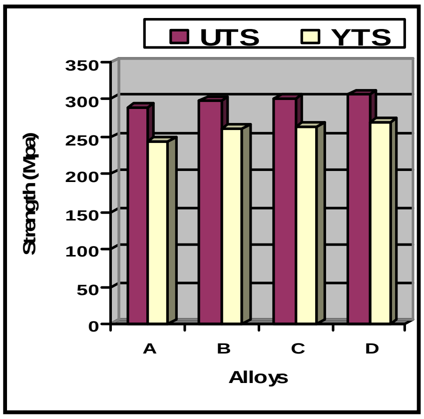
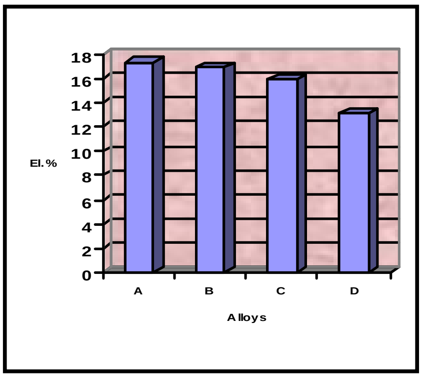
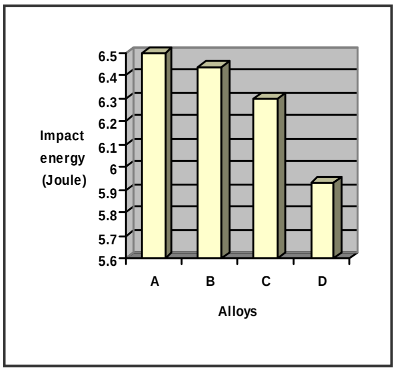


Figure (3): Vickers hardness number of alloys (A, B, C and D) after the post aging treatment at TMT_1 – practice



(4a)





(4b) (4c)

Figure (4): Effect of TMT_1practices of alloys (A, B, C and D) at peak hardness condition after post-aging treatment on;

a) Tensile strength(ultimate and yield), b) elongation percentage(EL%) c) impact energy.

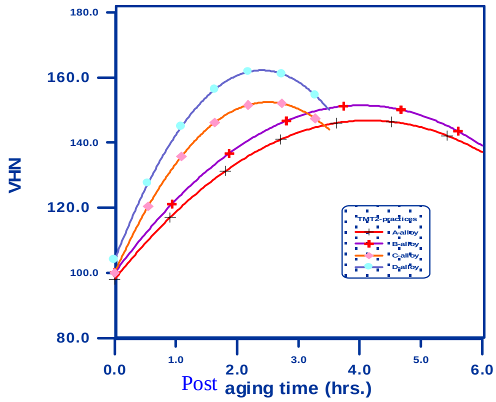
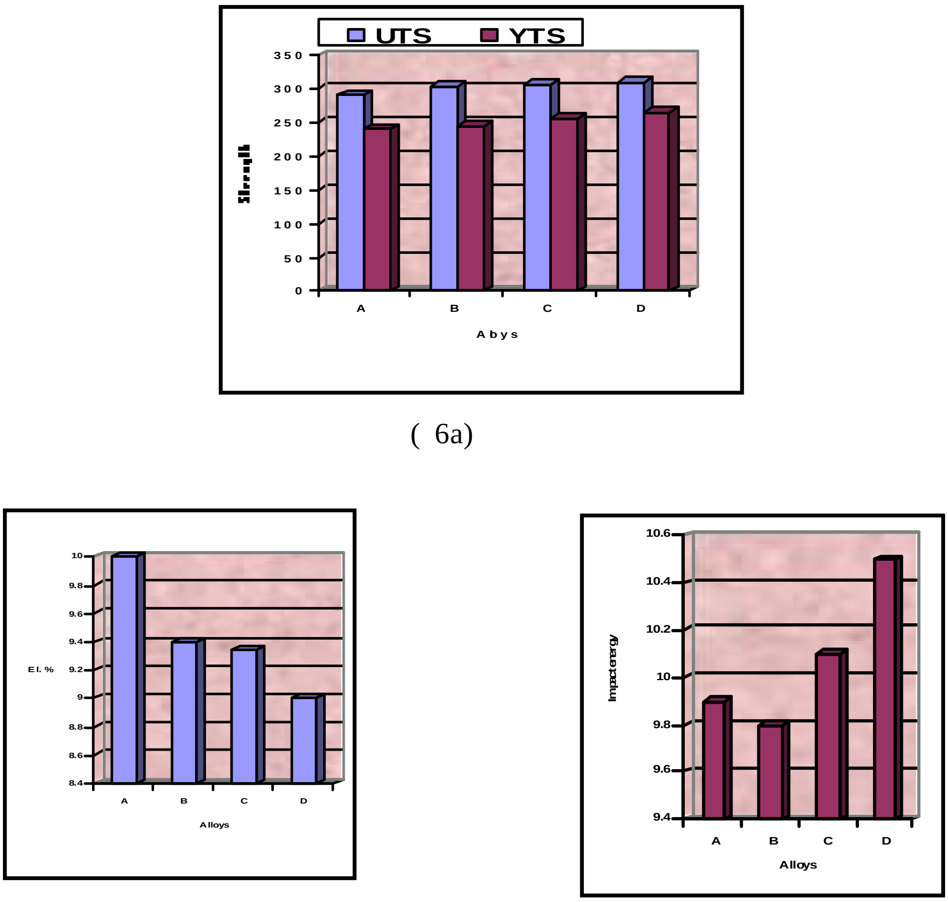


Figure (5): Vickers hardness number of alloys A, B, C and D after post-aging treatments at TMT_2 practices



(6a)

(6b) (6c)

Figure (6): Effect of TMT_2- practice of alloys (A, B, C and D) at peak hardness condition after post –aging treatment on;

a) Tensile strength(ultimate and yield), b) elongations percentage(EL%), c) impact energy.

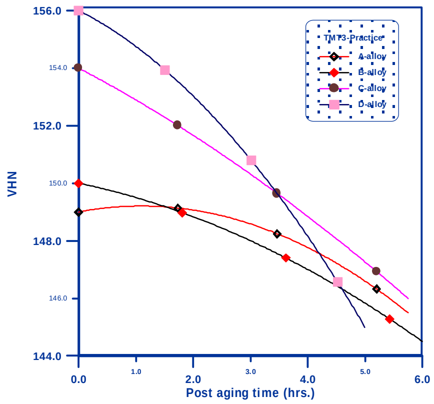
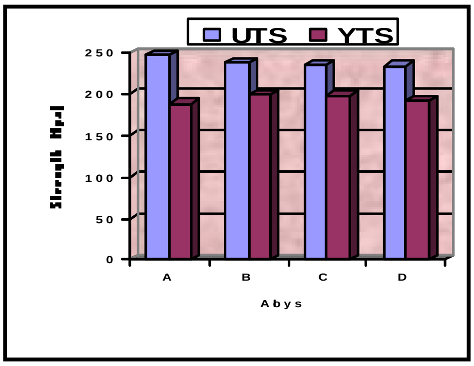
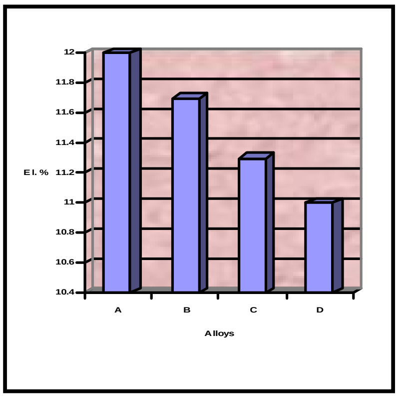
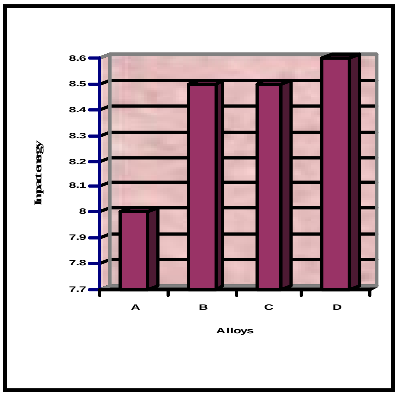


Figure (7) Vickers hardness number of studied alloy after the post-aging treatment at TMT_3 practices



(8a)





(8b) (8c)

Figure (8): Effect of TMT_3-practices of (A, B, C and D) alloys at peak hardness condition after post-aging treatment on;

a) Tensile strength(ultimate and yield), b) elongation percentage(EL%), c) impact energy.