Vol.(5),No.(1),(2017),10-15
MUTHANNA JOURNAL OF ENGINEERING AND TECHNOLOGY (MJET)
مجلة المثنى للهندسة و التكنولوجيا
Journal homepage:www.muthjet.com
Print ISSN:2572-0317, Online ISSN:2572-0325

Study of Differential Scanning Calorimetry on Phase Precipitation in Various Heat Treatments of AlZnMgCu Aluminum Alloys

Haider Tawfiq Naeem
Chemical Engineering Department., Engineering College, Al-Muthanna University

ARTICLE INFO

Received: 27/10/2016

Accepted: 6/3/2017

Keywords
Al_Zn_Mg_Cu alloys; Heat Treatment; Transition Phases; DSC

ABSTRACT

Powers compensation of the differential scanning calorimetry (DSC) have been utilized to revealed and analyze the phases of precipitations in the complex quaternary of Zinc (6.7wt. %) _ Magnesium (2.8 wt. %) _ Copper (1.8 wt. %) _Aluminum alloys manufactured by the process of the Semi-Direct Chill Casting. The casted Al-Zn-Mg-Cu alloys slabs were homogenizing at the different temperatures. Firstly under treatment 450°C for 2 hours follow by treatment 470°C for 1 day and then treatment at 480°C for 40 minutes, then all casted samples have quenched in cold water after each step. The treated homogenized Al-Zn-Mg-Cu samples that were underwent the artificial ageing at 120 °C for 1 day, and then retrogression at 180°C for 2400sec. and then re-ageing at 120°C for 1 day. The outcomes proved that the X-ray diffraction (XRD) analyses confirmed information of the DSC thermal analyses which were obtained through various heat treatments of the three samples AlZnMgCu alloy. The outcomes have explained that the DSC data are helping in understanding the changes the peaks of temperatures as well the enthalpy values for the forming and dissolution the equilibrium phase's and precipitation compounds within the artificial againg (with the pea-temper) and the Retrograession and Re-Againg-(RRA) treatments were conducting for Al_Zn_Mg_Cu alloy samples.

دراسة مسعر المسح التبايني على أطوار الترسيب في معاملات حرارية متنوعة لسبائك الألمنيوم (الألمنيوم-زنك-مغنيسيوم-نحاس)

الخلاصة

في هذه الدراسة تم استخدام معادلة القدرة التفاضلية لمسعر المسح التبايني (**Differential Scanning Calorimetry-DSC**) لكشف وتحليل مركبات الترسيب في الرباعية معقدةالالمنيوم- 6.7٪ الزنك -2.8٪ المغنيسيوم- 1.8٪ النحاس سبائك ، التي تنتجها عملية السباكة ذات التبريد السريع شبه المباشر.خضعت ألواح مسبوكة من خلائط آلامنيوم - الزنك- المغنيسيوم- النحاس الى معاملة المجانسة الحرارية عند درجات حرارة مختلفة .كانت هذه اولى تحت 450 درجة مئوية لمدة 2 ساعة يتبعها 470 درجة مئوية لمدة 24 ساعة واخيرا 480 درجة مئوية لمدة 40 دقيقة، بعدها تم اخماد العينات في الماء البارد بعد كل خطوة من عمليات التجانس. بالاضافة الى ذلك خضعت الواح السبيكة لمعاملة التعتيق الصناعي عند درجة حرارة 120 مئوية لمدة 24 ساعة ، وايضا على حرارة 180 درجة مئوية لمدة 40 دقيقة ثم إعادة التعتيق عند 120 درجة مئوية لمدة 24 ساعة خضعت كل العينات الى التبريد بالماء البارد بعد كل عملية حرارية. أثبتت النتائج أن معلومات تحليل حيود الأشعة السينية (**XRD**) تؤكد المعلومات الحاصلة من التحاليل الحرارية للمسعر المسح التبايني (**DSC**) لعينات الالمنيوم الخاضعة للمعالجات الحرارية المختلفة.من جانب اخر اوضحت النتائج ان البيانات الحرارية للمسعر المسح التبايني (**DSC**) ساعدت في فهم المتغيرات و قيم المحتوى الحراري لاطوارومركبات الترسيب عند التشكيل والانحلال الناتجة بتاثير التعتيق الصناعي عند 120 درجة مئوية وايضا عند عملية اعادة التعتيق في 120 درجة مئوية وثم عند 180 درجة مئوية تليها عند 120 درجة مئوية.

الكلمات المفتاحية
سبائك الألمنيوم-زنك-مغنيسيوم-نحاس ،المعالجة الحرارية ،الاطوار المنتقلة ،تقنية مسعر المسح التبايني

*Corresponding author.
E-mail addresses: dr.haider.ta@mu.edu.iq

DOI:10.52113/3/eng/mjet/2017-05-01/10-15

Introduction

The military, aero-space, transportation industries are more need to the novel materials have the highest physical-chemical and mechanical properties with the lower costs. Aluminium_Zinc_Magnesium_Copper Alloys of the 7000 series that considered are precipitations-hardend alloys with the master alloying of Zinc, Magnesium and Copper. principally, these series of Aluminium alloys have the highest mechanically strength and very good physical properties were appealing as a structural material to decreasing the weights [1, 2].

These properties are at most supervised by the solid solutions actions joined together with the heat processes. It can be reveal that the mechanism of precipitations through ageing of the supersaturating solid solution in those alloys are identified such as the follow sequence: firstly supersaturating solid solutions (SSS), then coherent GP zones, and then semi-coherent intermediate ή (MgZn2), finally the η incoherent stable η(MgZn2) [3]. Thus, the balance of properties of Al-Zn-Mg-Cu (7000) alloys can be optimized by microstructural adjustments via alloy compositional changes and various of heat treatment [4].

The heat processe that called retrogression and reageing-(RRA) was considering one of the main important the artificial ageing treatments to producing the precipitations within Al_Zn_Mg_Cu Alloys (7000) as well as which have the highe mechanical strength and stress corrosion craking more than the artificial ageing at the T6 tempering [3-4]. Differential scann calorimetriy (DSC) technicality was overall applied to take out accurate datum about the phases of precipitations transitions, in several zones besides recognize the solid state reactions joined with the dissolution of precipitates in heat treatable Aluminium-based alloys. Various analytical sketches are utilized for detection of kinetic parameters of the precipitation-phases transitions from the scan rate dependence of peaks revealed in the DSC curves. DSC is the powerful thermoanalytical technical for understand of the thermo-dynamics and kinetic energies of transition-phases which are changed.

At a current research-experimental, that considered the sample-(s) and reference-(r) materials that were heated linear in the furnace, Figure1 (a) and (b) display equipemnt and the schematics of the DSC device technique.

The samples in the test cruicible could underwent the transformaitions phases that aeither releasing (exothermics) or consume (endothermics) energies in the aform of the heat (enthalpies under constants the pressures). That heat was directe liken with the enthalpies expansion of the reference specimens which is known from independent previously experiment. In the continuous heating the DSC experimentes either the heat flux between two thermally jointed with references as well test samples is measured or by excess cold or heat the energies which is needful to recompense for the temperatures changing in the references pan [5-6]. With continuous heat DSC of quenched Aluminium-Alloys solid-state precipitations are given rise to peaks in the heat flux curve as a functions of temperature. The zone under the peaks, with reverence to the base line measured in the reference sample (R), is proportionally to the enthalpies change accompanying the precipitation process [7].

Regardless of numerous the DSC researches about the precipitations Aluminium Alloys (7000 series), neither informations of the phases of precipitation sequence follow nor the transitions of phase for the Aluminium_Zinc_ Magnesium_Copper alloys manufactured under a various parametres through in the present study, have completly yet.

Objective of this work is to provide an understanding of the phase transitions evolution of Al_Zn_Mg_Cu alloys made by (Semi_Direct_Chill Casting process) and affected via variations heat treatments. The precipitation kinetics parameters during the artificial aging treatment as well the Retrogression and Reageing treatment which are obtained using DSC technique with the combined of XRD analysis technique. In addition, the precipitation compositional of intermetallic through Al-alloys was acquired using XRD technique.

Experimental Procedures

The current study was used Aluminium Alloys (AA_7075) plates (13x 2.5x 1.3 cm). The nominals compositions of the current research aluminium alloy that recorded in Table 1. The chemical compositions analysiz was carried out using (Arc-Spark Spectrometer device).

Table 1: Chemical composition of studied Al-alloys (In weight percentage).

Materials	Amounts Weight %
Zinc	6.601
Magnesium	2.851
Copper	1.824
Iron	0.240
Silicon	0.076
Chrome	0.186
Titanium	0.028
Aluminium	Ball

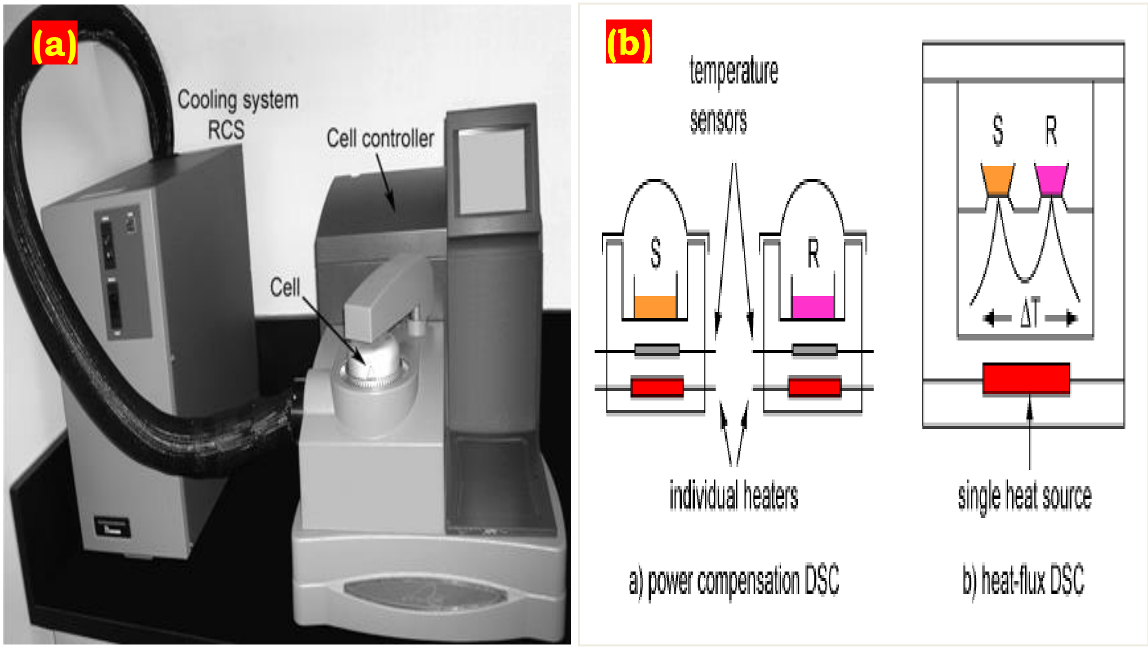


Figure 1: Show (a) DSC equipment picture with and (b) schematic illustration work techinque.

Al-alloys were re-melted in the graphite crucible at 870°C in electrical resistance furnaces. The spicements were manufactured using Semi Direct Chilling Casting applied in the cylindrical mold of iron steel, as more details about the methods of production by Naeem et al. [4,8]. Thereafter, Al_Zn_Mg_Cu alloys that were underwent the homogenizing treatements and follow ed by the artificial ageing at (the T6-temper) then the retrogression and re-ageing, as mentioned more information [4]. For the testing of differential scanning calorimetry (DSC), heat flux type Shimadzu_DSC-50 was utilized. The samples of DSC were cut in shape of discs from the plates which had been heat-treated. The DSC-samples were fabricated from the Al-alloy plates to discs 5mm in diameter and 1mm in, height with average mass of about 10 mg. The DSC experiments was performed at heating rate 10 °C/min. in normal atmosphere. The X-ray diffraction analysiz was used (XRD-6000, SHIMADZU) as detailed in recent [8].

Results and Discussion

In order to know the phenomena of the nucleation and growth of precipitation phases in alloys, through heat treatments, were investigated by DSC analysis. A DSC curves for Al-samples under the different conditions (the as quenched, T6-temper and trated RRA) is presented at figure 2. That could be notifed that the peak A represened to the dissolution of Guinier-Preston zones (GP_Z) related to the as-quenched curve as well the reversion of the GP zones and fine η′-(Mg_2Zn_{11}) phase related to the T6 and RRA treatments. The peak B relating to the transformations from the GPz and the intermediate (η′) phase to an equilibrium phases η-($MgZn_2$), which is disappear in the Al_Alloy samples after the RRA process wiht the as quenched condition.

At increase the heating rate, the peak C displays the dissolution of residual the GP zones and η′phase precipitates. The stability of peaks D and E that are attributed to the creation of the phase (T ($Al_2Mg_3Zn_3$) and thereafter (S) phase (Al6CuMg4) between the tempratures of 230°C - 380°C, as reported [3, 8]. The peak F which corresponding to the dissolution of S phase, in addition to effects of peak G is ascribed to melting of non–equilibrium Al/Al Zn MgCu phase at 479 °C, which is disappear through T6 temper and RRA (due to series of solution heat treatments have been carried out on them led to dissolved α-Al/ (AlZnMgCu) phases into the matrix completely).

The peak H (494.7°C) melting reaction was observed, that clarify transformation from the eutectic phases into higher melting point the Al_2CuMg phase, which applied in sample base Al_alloy within the T6 but that disappear in RRA process.

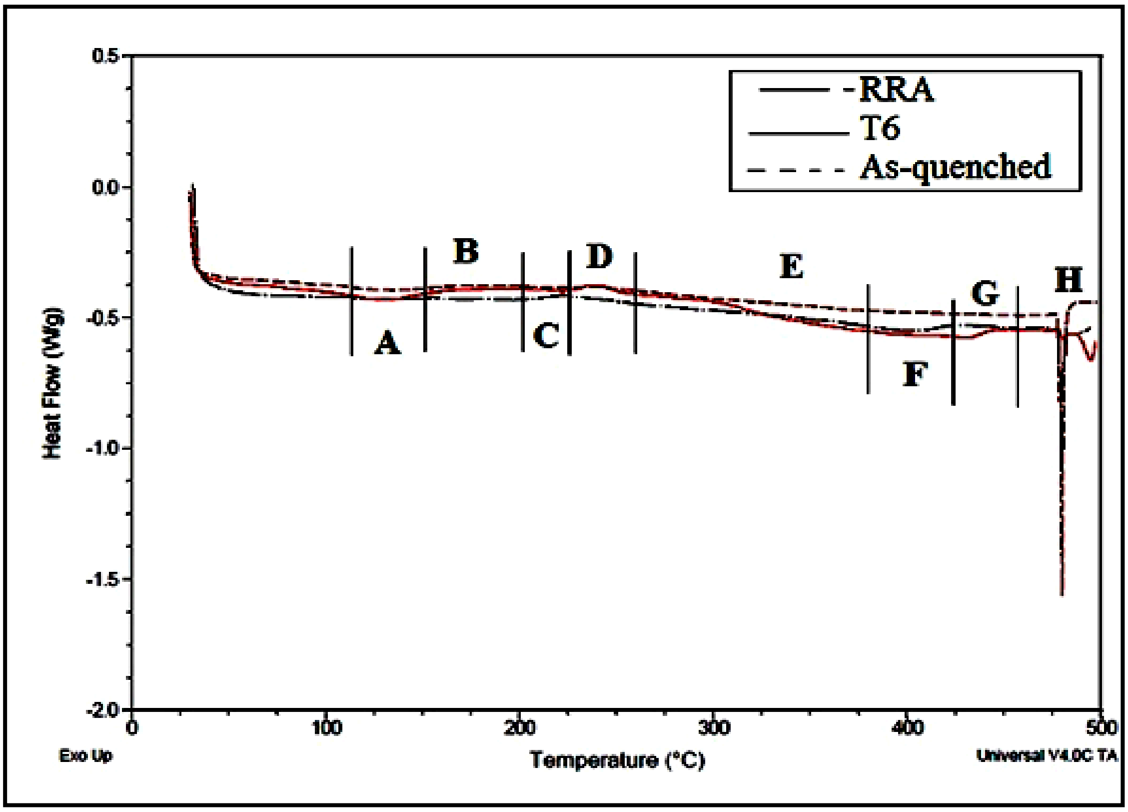
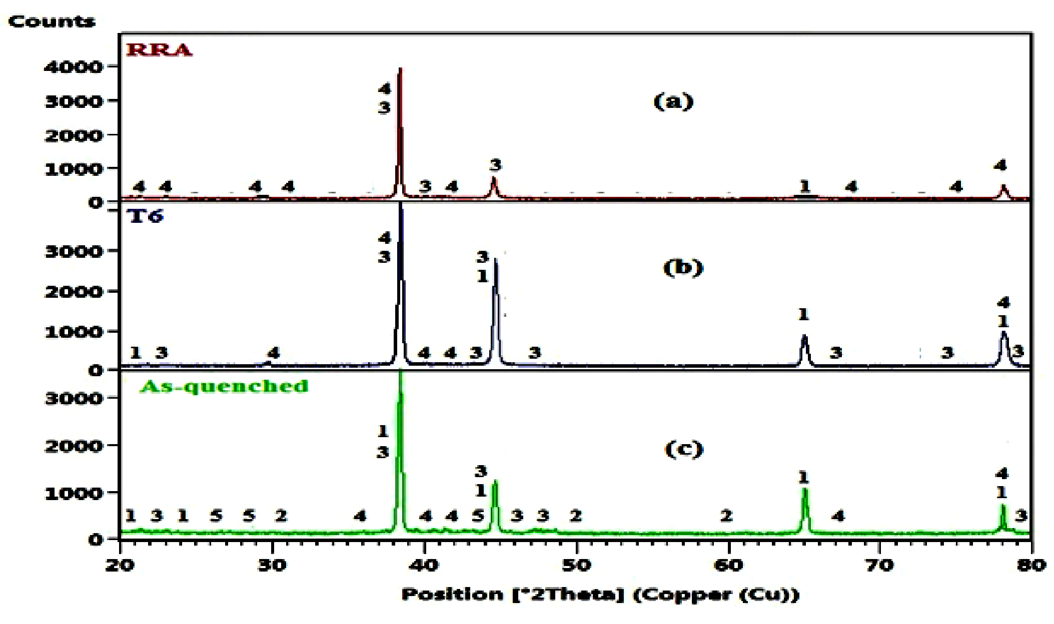


Figure 2: DSC curves of three Al-7075 alloys underwent the quenched, the aging treatment at T6 and the RRA process.

It could be explaining the differences of the DSC curves among the varsion conditions for Al-Zn-Mg-Cu alloy samples through the helping DSC data as show in Table 3 that included Tp denotes the peak temperature and ΔH denotes enthalpy value. The areas under the DSC curves related to the volume fractions of the phases, which calculating the qualitatively using its enthalpy values. The peaks temperature of given reactions are dependent on the phase composition under the same conditions. It observed that enthalpy T6 at (peak A) about -2.5776 J/g higher than the RRA treatment; it is supposed that there are the extra solution atoms after the ageing at T6-temer. The enthalpy of peak B (RRA) higher than the T6 temper through this stage, which is process for the consuming Zinc and Magnesium solute atoms availabled in the matric, thus the enthalpies majority depend on the amount of Zn and Mg remained [2, 9, 10]. Moreover, it could be noticed that the enthalpy of peak G in as-quenched curve which is -7.11J/g, higher than both of the T6 and RRA, such increase in enthalpy due to found the non-equilibrium solidification eutectic during as quenched, which disappear in T6 and RRA, that attributed to series of solution heat treatments have been carried out on both of T6 and RRA led to dissolved Al/AlZnMgCu phases into the matrix completely. The various X-ray diffraction patterns for Al_samples undergo the as quenched with the T6 temper and the RRA process in Figure 3. It observed as quenched alloy is composed of α-(Al) and secondary phases which were mainly; T-$AlMg_4Zn_{11}$, S-Al_2CuMg, $Al_{23}CuFe_4$,η-$MgZn_2$ and η′-Mg_2Zn_{11} phase. The prime precipitation in the matrix of Aluminium-7000seris-alloy are the GPzs and η′-Mg_2Zn_{11} phase underwent the artificial ageing at T6 temper and the XRD plot for it detect the presence growing up in the phases of η and η′ which precipitate. The X-ray diffiraction plots of samples at the RRA treated, the phases that peaked high were $MgZn_2$ and Mg_2Zn_{11}. The GPz plentifully and phase (η′) that are re-dissolved within the early soon stages of the Retrogression tread.The lot of nucleus GPz and the η′ phase where can raise re-precipitations in the reageing tread and they were raising up, mentioned in previous studies [8, 11]. Table 3: Temperature Peak (Tp) and Heat of reaction obtined from DSC curves for Al-samples alloy underwent various heat treatment.

Table 3: Temperature Peak (Tp) and Heat of reaction obtained from DSC curves for Al-samples alloy underwent various heat treatment.

Peaks	Details	As-quenched	T6	RRA
A	Tp(°C)	135.62	130.54	155
	ΔH(J/g)	-2.367	-2.5776	-2.523
B	Tp(°C)	250	175	222
	ΔH(J/g)	-2.4103	-2.1402	-2.508
C	Tp(°C)	350	223.3	270.56
	ΔH(J/g)	-2.8321	-2.3922	-2.2911
D	Tp(°C)	0	238	290
	ΔH(J/g)	0	-2.2608	-2.201
E	Tp(°C)	0	299.13	320
	ΔH(J/g)	0	-2.6214	-2.54
F	Tp(°C)	0	431.42	402
	ΔH(J/g)	0	-3.456	-3.2784
G	Tp(°C)	479.18	479.18	479.18
	ΔH(J/g)	-7.11	-3.381	-3.381
H	Tp(°C)	0	494.74	0
	ΔH(J/g)	0	-3.9666	0



1-Al, Al Mg_4Zn_{11} 2- Al_2CuMg 3-Mg_2Zn_{11} 4-$MgZn_2$ 5-$Al_{23}CuFe_4$

Figure 3: XRD for Al_Zn_Mg_Cu samples Alloys underwent (a) the Retrogression and reaging, (b) Ageing at T6 and (c) the As quenched casting.

Conclusions

1. Regarding the DSC observations gave the description the peaks of heat effect and thermic parameters of the forming and disintegration for the GP zones and the precipitation phase such as η′-(Mg_2Zn_{11}) , η-($MgZn_2$), and other equilibrium phases within the Al-samples after applied the ageing at T6 peak with the RRA treatment.
2. Additionally, the microstructures observations of the heat treated Al-samples (with the XRD technique) revealed the existence of various of the main precipitations in the matrix are Guinier-Preston zones, intermediate phases as well the main phases of α-(Al), T-($AlMg_4Zn_{11}$) and S-(Al_2CuMg).

Acknowledgments

The author thanks University Malaysia Perlis (Malaysia) and Muthanna University (Iraq) for the support provided to complete this work.

References

1. P. Lang , T. Wojcik, E.Karadeniz , A. Falahati, 2014 “Thermo-kinetic prediction of metastable and stable phase precipitation in Al–Zn–Mg series aluminium alloys during non-isothermal DSC analysis”,Journal of Alloys and Compounds 609 pp. 129–136.

2. X.M. Li and M.J. Starink2012, “DSC Study on Phase Transitions and Their Correlation with Properties of Overaged Al-Zn-Mg-Cu Alloys”, Journal of Materials Engineering and Performance Volume 21(6) pp.977.

3. Haider T. Naeem, K. S. Mohammad, K. R. Ahmad, 2014 “The Effect of Microalloying of Nickel, RRA Treatment on Microstructure and Mechanical Properties for High Strength Aluminum Alloy”, Advanced Materials Research Vol. 925 , pp 253-257.

4. Haider T. Naeem, K. R. Ahmad, K. S. Mohammad, A. Rahmat, 2014 “Evolution of the retrogression and reaging treatment on microstructure and properties of Aluminum alloy (Al-Zn-Mg-Cu)”, Advanced Materials Research Vol. 925 , pp. 258-262.

5. S.K. Panigrahi, R. Jayaganthan, V. Pancholi, M. Gupta,2010, “A DSC study on the precipitation kinetics of cryorolled Al 6063 alloy”, Materials Chemistry and Physics 122 pp. 188–193.

6. Bershteĭn, Vladimir Abramovich, and Viktor M. Egorov. 1994"Differential scanning calorimetry of polymers”, Thesis, Humboldt Universitat ZU Berlin, Prentice Hall,.

7. E. Hersent, J.H. Driver, 2010 “Modeling Differential Scanning Curves of Precipitation in Al-Cu-Mg”, Scripta Materialia, Vol. 62, No. 7, pp-455-457.

8. Haider T. Naeem, Kahtan S. Mohammed, 2013 “Retrogression and Re-Aging Of Aluminum Alloys (AA 7075) Containing Nickel”, Digest Journal of Nanomaterials and Biostructures Vol. 8, No. 4, ,p p. 1621 – 1632.

9. W.X. Shu, L.G. Hou, J.C. Liu, C. Zhang, F. Zhang, J.T. Liu, L.Z. Zhuang, and J.S. Zhang, 2015 “Solidification Paths and Phase Components at High Temperatures of High-Zn Al-Zn-Mg-Cu Alloys with Different Mg and Cu Contents”, Metallurgical And Materials Transactions A Vol. 46A,pp.5375.

10. X.-M. Li and M. J. Starink,2001, “The Effect of Compositional Variations on the Characteristics of Coarse Intermetallic Particles in Overaged 7xxx Al Alloys”, Mater. Sci. Techn, , vol. 17, pp.1324-28.

11. Haider T. Naeem, Kahtan S. Mohammed, Khairel R. Ahmad, and Azmi Rahmat, 2014 “The Influence of Nickel and Tin Additives on the Microstructural and Mechanical Properties of Al-Zn-Mg-Cu Alloys”, Advances in Materials Science and Engineering Volume, Article ID 686474,pp. 10.