Preparation of Al-12si Foam Using Liquid Technique

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Abstract

This research aims at foaming an Al-12Si alloy by using $CaCO_3$ powder as a foaming agent added to the agitated melt in different weight percents.

The foaming process was carried out at different foaming temperatures of 700C°, 750 C° and 800C° for 5, 10 and 15 minutes using different foaming agent percents (0.5, 1, and 1.5) wt%. The metallic foam had been characterized by microstructure examinations, density and porosity and compression tests.

It was found from the results that there is an ability to produce metallic foams based on Al –Si alloy by using $CaCO_3$ as a foaming agent without the need for adding ceramic stabilizing materials which are always added in a high percent. The mach inability problems caused by the presence of these materials can therefore be avoided.

It was also pointed out that the porosity of metallic foams fabricated in this work increased as the foaming agent percent increases within the range used (0.5% - 1.5 %) and the preferred amount is 1%, where the optimum foaming time is 10 minutes at a foaming temperature of 750 C°.

Keywords: metal foam microstructure Al-Si alloy

تحضير رغوة الالمنيوم –سيليكون12بلستخدام تقنية الحالة السائلة

الخلاصة

يهدف البحث انتاج رغوة معدنية من سبيكة الالمنيوم – 12سليكون وذلك باضافة كاربونات الكالسيوم كعامل ترغية الى المعدن المنصهر اثناء عملية الخلط وبنسب وزنية مختلفة. ثم اجراء عملية الترغية عند درجات حرارية هي (800،700،750)م˚ وبازمان ترغية هي (15، 5،10) دقيقة وباضافة نسب مختلفة من عامل الترغية هي (0.5،1.5،1)% . تم توصيف الرغوة المعدنية الناتجة باستخدام الفحوصات المحهرية وفحوصات الانضغاط ، كما تم حساب الكثافة والمسامية. من نتائج التجارب وجد ان هنالك امكانية لانتاج رغوة معدنية من سبيكة الالمنيوم-12 سليكون باستخدام كاربونات الكالسيوم كعامل ترغية بدون الحاجة الى اضافة دقائق سيراميكية مثل كاربيد السليكون والتي تضاف بنسب عاليةلغرض استقرار الرغوة المعدنية. وبذلك يمكن تقليل المشاكل الناجمة عن صعوبة تشغيل الرغوه الحاوية على نسب من Sic لوحظ من النتائج ان نسبة المسامية للرغوة المعدنية المنتجة تزداد مع زيادة نسبة اضافة عامل الترغية. وان افضل نسبة مسامية تم الحصول عليها عند اضافة 1% كاربونات الكالسيوم وزمن ترغية 10 دقائق وبدرجة حرارة 750م˚.

Introduction

Metallic foam can be defined as a metallic material with a cellular structure which has an interesting combination of physical and mechanical properties, such as light

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weight in conjunction with high stiffness, or ultra low weight combineding with high thermal insulation withstand elevated temperatures [1]. Metallic foam can be produced with open or closed cells depending on the type of application field [2, 3, 4].

The unique properties of foams promise a variety of applications in vehicle design, ranging from light-weight construction, impact energy absorption to various types of acoustic damping and thermal insulation [5]. The development of production methods

Calcium   carbonate   powder (CaCO₃)

Calcium carbonate powder was used as a foaming agent, supplied by (LOBA CHEM. INDIA), the purity of powder is (99%) with a particle size of (< 10µm).

Aluminum -12 Silicon powder

Aluminum -12 silicon powder was used as a distributer of the foaming agent, Al-12Si powder was prepared by ball milling of Al-12Si alloy, with   particle size ranging between (100 -200) µm.

2- Experimental Procedure

Preparation of Foaming Agent Mixture

Foaming agent mixture was prepared by mixing Calcium carbonate powder and Aluminum - 12silicon powder as a weight percent ratio (1:2). The mixture was mixed in an   electrical   mixer at a rotation speed of (100) rpm for (5) minutes.

Metallic foam fabrication

In order to get a desired porosity of the fabricated samples, the metallic foam is prepared in two stages. In this process, the foam precursor is fabricated first and the metallic foam is then issued by the backing stage.

for metallic foams has given rise to renewed interest in these materials [6]. Various methods for making such foams are available, include liquiding, solid, vapor deposition, and electro deposition techniques.

2-Experimental work
Materials Used
Aluminum -12Silicon alloy

Aluminum -12Silicon alloy is used as a base foaming material, due to the wide range of applications of this alloy. Table (1) shows the chemical composition of the alloy used in this work.

First   Stage   (foam   precursor fabrication)

Foam precursor was fabricated by melting (2kg) of Aluminum - 12Silicon   alloy in a steel crucible in an electrical resistance furnace at (860ºC). The temperature difference between the crucible and furnace was (30ºC ± 2ºC) and the crucible temperature was displayed by a digital reader connected to a (Ni-Cr) thermocouple, fixed to a steel sheet under the crucible. When the alloy melting was completed and the furnace temperature reached (860ºC) , the furnace was turned off   and agitation started at a rotation speed of (1200)   rpm   . The agitation continued till the crucible temperature reached (700ºC). The   mixture   of foaming agent was then gradually added in a weight percent of (0.5%) of the melt weight while agitation was kept till the temperature of the crucible reached (600ºC) . At this moment , the agitation was stopped and   the   melt was poured into a rectangular steel mould with dimensions of (3 x 15 x 15) cm³ . The mould was then cooled down by a jet of air, and the precursor was removed from the mould. The volume and mass of the precursor was accurately

calculated. Figure (1) shows the produced precursor. The density of the precursor was calculated using the following equation:-

$$\rho = m/v \qquad ------- (1)$$

Second Stage (Backing of the precursor)

The backing process of the foam precursor was carried out by using a heat treatment furnace type (Carbolite DT 109). A portion of precursor which has a specific size (the size depends on the porosity required) located in the backing die , the backing die is a steel cylinder (50) mm external diameter , 42 mm internal diameter and 70 mm height) the two sides of die were sealed with two steel sheets, which were fixed together by four bolts . Figure (2) shows the backing die.

The backing die was placed inside the furnace, heated to (750 ºC), for 10 minutes. The furnace was then turned off and the die was removed and cooled down by an air jet . The foam sample produced was then removed and sectioned into the required dimensions and prepared for the following testing. Figure (3) shows the path of process steps.

Characterization and Testing Of Metallic Foam

In order to characterize the metallic foam produced in the present work several tests were performed, these tests are:-

Porosity Evaluation

The porosity of the metallic foam resulting from one stage process was calculated from the density of the samples. The samples in this case have an inhomogeneous shape with closed outer skin and internal closed cells, as shown in figure (4). The sample weight was measured by an electrical balance of four digits type (TP-214);

the accuracy of the measurement method is about (0.0001gr). The samples were then coated with a thin layer of grease to avoid penetration of water through any pore that may be present. The samples were immersed into a scaled vessel containing water. The volume of displacement represents the sample volume. Relative density of metallic foam (ρ^*) can be calculated using the following equation:-

$\rho^* = \rho_f / \rho_s$, where ; ρ_f is the density of foam (gr/cm³) , ρ_s represents the solid density (gr/cm³) So the porosity of metallic foam can be calculated using the following equation:-[16]

$$\text{Porosity } (\%) = 1 - \rho^* \qquad ----- (2)$$

Microstructure Evaluation

Microstructure evaluation was carried out on metallic foam samples using an optical microscope with a digital camera with different magnifications in order to study and characterize the metallic foam cell size , porosity and their distribution . The samples were ground by Silicon carbide papers of (500, 1000, and 1200) grade, then polished with a cloth, and etched with (20%) NaOH for (30) seconds.

Compression Test

The compression test was done on the samples prepared by using a two stage route with different porosities (63%, 74%, 82%), three samples for each porosity. The test was carried out using the universal testing machine type (INSTRON 1195), a cross head speed (0.5) mm/min., and (1ton) load .The samples were sectioned into dimensions of (1.2 x 1.2 x 2.4) cm³.

Energy absorbed by the metallic foam was calculated through measuring the areas under the stress –

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strain curves at a strain range from (5) to (25) percent.

3 - Results and Discussion
Microstructure Examination

Microstructures of metallic foam samples were examined in order to study and to characterize the behavior of metallic foam cell wall , foam cell size , porosity and its distribution and to explain how they are affected by foaming variables i.e (temperature , time and foaming agent percent).

Figure (5) shows the microstructure of as cast Al-12Si alloy. The microphotograph indicates that the microstructure contains α-Al (white), primary Silicon (light grey) and eutectic. While figure (6) represents the microstructure of foam precursor containing 1%$CaCO_3$ revealing some particles that were dispersed throughout the cast as well as empty irregular pores with different sizes. The appearance of the fine bright particles may be attributed to the Aluminum oxide which was formed during the prior agitation of the melt [7]. The irregular pores were the sites of $CaCO_3$ powder that was removed during the preparation of the sample surface for the microstructure examination.

Figure (7) shows a microphotograph of the foam cell wall of the metallic foam with 1%$CaCO_3$ at 700˚C and 750˚C .It appears to be as a texture decorated with precipitates. These precipitates may represent various mixed oxides of aluminum, calcium, such as Al_2CaO_4 or oxides mix Al_2O_3+CaO, or inter metallic compounds such as Al_4C_3 which were formed during the foaming process by the thermal decomposition of $CaCO_3$ as clarified in equations (3) ,(4) and (5)[7].

$$4CaCO_3 =4\ CaO +4\ CO_2 \quad ---- (3)$$

$$2Al +3CO_2 =Al_2O_3 +3CO ---- (4)$$
$$8Al + 3CO_2 = 2Al_2O_3 + Al_4C_3 - (5)$$
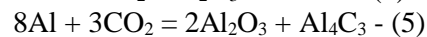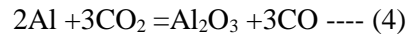
The microstructure examination also revealed that the inter lamellar eutectic distance is refined at adjacent regions near the pores.

These phenomena may be attributed to the mechanical waves which resulted from the bursting of some gas bubbles, which enhance the nucleation process of the eutectic phase [8] or by breaking the eutectic plates by the agitation of melt. The precipitates also concentrated on or near the solid/air interface, and seem to be slightly distributed over the cross section of the cell walls as shown in figures (8) and (9).

Foam cells start as spherical gas bubbles with a regular shape, during the formation of Skelton foam. Some of these cells retain their shape till the solidification is accomplished, and appear as circular pores, as shown in figure (9), which presents the microstructure of metallic foamed with

(1%) $CaCO_3$ at (750˚C). On the other hand, some of foam cells lost their spherical form and appeared in different irregular shapes, as shown in figure (10) which represents the microstructure of metallic foamed with (1.5%) $CaCO_3$ at (750˚C).

The presence of regular small pores as shown in figure(9) may be related to pores that were created as a result of the carbon dioxide formation , then when this gas was reduced to carbon monoxide by the oxidation of Aluminum [7]. The final content of the foam cell was carbon monoxide gas with a low partial pressure [9] and thin Aluminum oxide layer covering the internal cell surface, which in turn stabilizes the shape of the foam cell.

The foam cell shown in figure (10) looks irregular because the pores

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had accumulated to form irregular cell shapes or as a result of the large expansion of the internal foaming gas, which leads to formation large bubbles having a high surface area which were easily buckled [10].

Effect of foaming variables on foam porosity

Effect of foaming time

During the fabrication of metallic foam with different foaming times (5, 10, 15) minutes, the results revealed that foaming occurs after 5 minutes. The foamed samples appeared slightly convex on the top surface as shown in figure (11), which may be related to the low foaming agent decomposition during this relatively short time [11,12].

The sample foamed within 15 minutes has failed; this may be due to the continuous heating for a relatively long time that leads to decrease in melt viscosity and then the stability of the cell wall of the metallic foam which results in complete decay of foam feature [10] as shown in figure (12).

Depending on the previous results, 10 minutes foaming time was selected as a preferred foaming time for the fabrication of Aluminum – Silicon samples foamed with calcium carbonate powder.

Effect of foaming temperature.

The effect of foaming temperature on the porosity of the foamed samples was studied in the range of (700C°-800C°). It was found, as shown in figure (13) that porosity increases as temperature increases i.e. porosity was found to be 53% at 700C° and increased to reach its maximum value 58% at range of (750-800)C° and at 0.5% of $CaCO_3$, while at 1% of the foaming agent $CaCO_3$, the results in figure(13) revealed that porosity increased from 65% to 76% as temperature increased from 700°C to 750C° , then a clear decrease in porosity value was observed as temperature increased to 800C° , and became 69%. With further increase in foaming agent i.e. 1.5% of $CaCO_3$, the behavior of porosity value change was similar likewise for the addition of 1% of $CaCO_3$, i.e. porosity increased from 72% at 700C° to 75% at 750C°, decreased to 72.5% again at 800°C. From this, we can easily conclude that the maximum porosity obtained was 76% at 750°C, using 1% of foaming agent $CaCO_3$, while the minimum porosity obtained was 53% at 700°C, using 0.5% of foaming agent $CaCO_3$. The dissociation of limestone ($CaCO_3$) leads to the formation of lime (CaO) and carbon dioxide (CO_2) gas which acts as a foaming catalyst and for that large porosity are formed with the increase of $CaCO_3$ content, because of an incremental increase in gas bubbles.

Also the formation of $CaAl2O_4$ phase as well as CaO will be diffused through the melt and enhance the stabilization of the metallic foam, as long as the viscosity of the melt increases with increase in the foaming agent ($CaCO_3$). At low temperature i.e. 700°C , partial dissociation of $CaCO_3$ [14,15] , in other mean , the decomposition of foaming agent is not accomplished , for that viscosity of the melt is lower than at 750°C , which in turn will affect the stability of the resultant metallic foam .

The results coincide with macrostructure observation which revealed the appearance of massive agglomerates of un-decomposed $CaCO_3$ as shown in figure (4).

The cross section of metallic foam at higher temperatures and higher CaCO3% (750°C, 1%) respectively, on the other hand, revealed the

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absence of $CaCO_3$ because of the complete decomposition of the agent accomplished as shown in figure (4).

Compression test

Metallic foam undergoes plastic deformation with a typical yielding when it is exposed to an external compression load. Figure (14) shows stress – strain curve for metallic foam samples with relative densities of 0.37, 0.26 and 0.18 foamed at 750°C for 10 minutes by using a foaming agent of 0.5% wt.

It seems that stress increases linearly to a value of 15 Mp in the range of strain from (0 – 5%), yet stress remains constant, while the strain largely increases till 23% strain. After this point the stress proportionally increases with a small amount of strain change.

The explanation of this behavior of curve indicates that foam sample had been deformed elastically in the first region (0-5%) strain and then plastic deformation started at 15MPa and continued with stress constancy. This type of metallic foam deformation continued throughout the region to (5%-23%) strain. Finally , the proportional increase of stress with a few percents of strain change (region three) was due to the fact that the foam samples behave as a solid matter since the foam cells were completely destroyed and densification had occurred [16,17]. The same behavior of metallic foam samples with relative densities of 0.26 and 0.18 were observed except that the values of stresses, strains and strain ranges had changed depending on the relative density of samples.

Furthermore, there was oscillation of the flow stress during plastic deformation regions. This could be due to the non homogeneity of high porosity samples and / or weakness of cell wall.

The results obtained from the compression test pointed out that the flow stress of continuous deformation of metallic foam was a function of relative density. Figure (15) shows that the maximum flow stresses at higher relative density and the stress dropped when relative density had decreased.

The energy absorbed by the metallic foam samples which had been calculated from the area under the stress – strain curves is presented in figure (16).

It can be seen from the figure that energy absorbed by the metallic foam samples was reduced with the decrease of relative density, as the cubic meter of foam pointed that a relative density of 0.37 had been absorbed 3 MJ and 2.5 MJ absorbed by a cubic meter of a foam with a relative density of 0.26, while the metallic foam of relative density 0.18 had been absorbed 0.8 MJ.

If the weight of metallic foam is considered, the measured values of energy absorbed per Kg of metallic foam are listed in table (2).

As appears in the table that metallic foam with a relative density of 0.26 had absorbed higher amount of energy than the metallic foam of 0.37 relative density, which means a good combination of low weight and high energy absorption so the metallic foam with (ρ*= 0.26) was the best choice as a mechanical energy absorber compared with the other samples which have been fabricated in this work.

4- Conclusions

From the present work, the main conclusions which can be drawn are:-
1-Stable metallic foam can be produced using $CaCO_3$ powder to

foam Aluminum – 12Silicon alloys without the need of adding ceramic materials.

2-Awide range of porosity percentages can be acquired by using the two stage route.

3-Porosity of Al-12Si foam increased with the increase of $CaCO_3$ percent within the range of (0.5 -1.5) % and the preferred percentage of $CaCO_3$ was 1%wt, with foaming temperature of 750°C and 10 minutes foaming time.

Referances

[1] **J. Banhart** ,“ Manufacture , Characterization and Application of Cellular Metals and Metal Foams”, Progress in Materials Science, vol.46 2001, pp. 559–632.

[2] **E. Amssterdam , J.Hosson , P.R.Onck** "on the Plastic Collapse stress of Open– Cell aluminum Foam ". Scripta Materialia , Vol 59, No 6 September 2008 PP 653-656 .

[3] **S. Esmaeelzadeh , A.Simchi and D.Lehmhus** ,“ Effect of Ceramic Particle Addition On The Foaming Behavior ,Cell Structure And Mechanical Properties of P/M AlSi7 Foam” , Materials Science and Engineering , Vol. 424 , 2006 ,pp.290-299.

[4] **A.Oudka , F.Mreno, J.Banhart "Structure and** Distribution of Oxides in Aluminum Foam" Acta Materialia , Vo 156, No 15, September 2008 PP 2990-4001.

[5] **J.Banhart** ,“ Aluminum Foams For Light Vehicles ” , International Journal For Vehicle Design , Vol.37 , No.2 , 2005 , pp.114-125.

[6] **D.Lehmus,J. Banhart**,“ Properties Of Heat –Treated Aluminum Foams ”, Materials Science And Engineering , No. 349 , 2003 , pp.98-110.

[7] **V. Gergely,D.C.Curran and T.W. Clyne**, “The Foam carp

rocess” Composites science and Technology , No.63 , 2003 ,pp. 23.

[8] Z.Kadhim ,“prediction of shrinkage cavities in Aluminum-Silicon sand casting” , PhD thesis , university of technology , 2007 ,pp.80.

[9] **J.Banhart** ,“Manufacturing Routes For Metallic Foams”, Journal Of Materials , Vol.52 , No.12 , 2000 ,pp.22-27.

[10] **I.Duarta, J.Mscarenhas, And A. Ferreira** “The Evaluation of Morphology and Kinetics during the Foaming Process”, Engineering Materials, Vol. 230, 2002, pp.96-101.

[11] **S.Cox ,G.Bradley And D. Weaire** ,“Metallic Foam Processing From The Liquid State ” ,Applied Physics Journal , September ,2000 ,pp.1-9.

[12] **S. Budavari**, “The Merck Index”, Professional Hand Book, 1995, pp.272.

[13] **L. Peroni, M.Avalle, M.Peroni "The Mechanical behavior of aluminum Foam Structure in Deferent Loading** Conditions" International Journal of Impact Engineering, Vol 35 No7, July 2008 PP 644-658.

[14] **T. Yan-Jun; L. You-Ming, S. Jing And P. Zhi-Dong**,“ Structural Characterization and Thermal Decomposition Behavior of Micro sized and Nano sized $CaCO_3$” , State Key Laboratory of Pulp and Paper Engineering , University of Technology , South China , 2007.

[15] **J. Liu And J. Zhang , “Assessment Of The Apparent Activation Energies For Gas/Solid Reactions-Carbonate Decomposition” ,** Journal Of University Of Science And Technology Bejing , Vol.10 No.2 2003 ,P.25-29.

[16] **C. Yu** and **J. Banhart,**
   "Mechanical Properties of Metallic
   Foams", meat foam con.,USA
   ,1998.
[17] **I. Hall, M.Guden and C. Yu**,
   "Crushing of Aluminum Closed

Cell Foams" Script Materials,
No.43, 2000, pp.515-521.
[18] **Y.Cegel** "Heat Transfer", 2nd Ed.
   2005, pp.64-65, McGraw-Hill,
   India.

Table (1) the chemical composition of the aluminum silicon-12 alloy.

% Si	11 – 12
%Fe (max.)	1
%Cu	1.75-2.5
Mn (max.)%	0.5
%Mg(max.)	0.4
Ti(max.)%	0.15
Zn(max.)%	0.7
%Al	Bal.

Table (2) Energy absorbed per volume and per mass of foam samples

ρ^*	Energy absorbed per volume (MJ/m³)	Energy absorbed per mass (KJ/Kg)
0.37	**3**	**3.06**
0.26	**2.5**	**3.62**
0.18	**0.8**	**1.67**

Figure (1): The precursor produced in the work	**Figure (2):** The Backing Die.

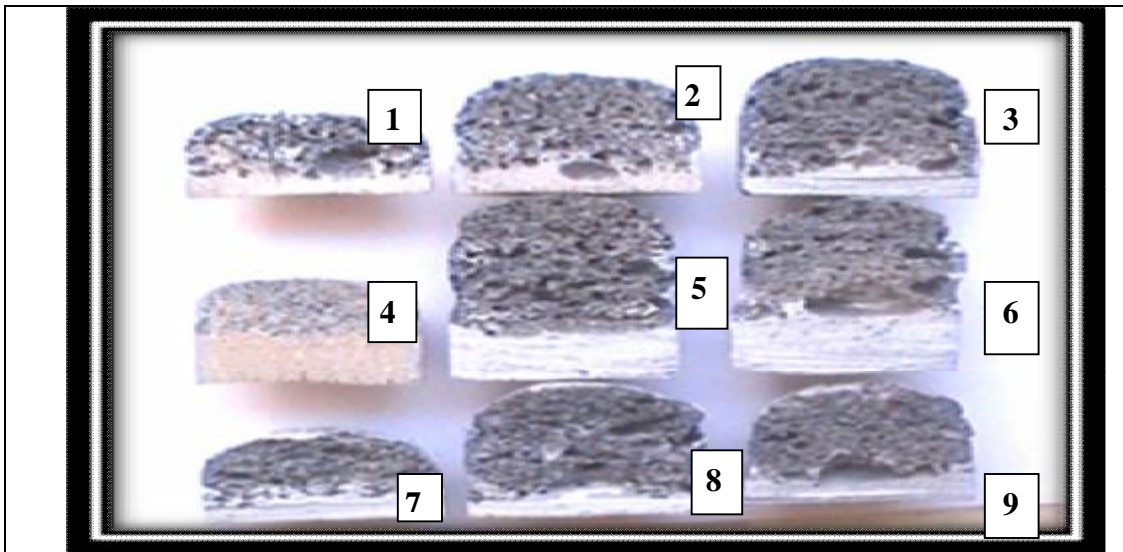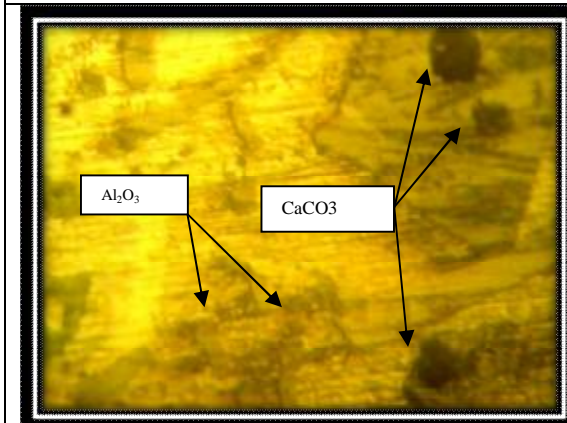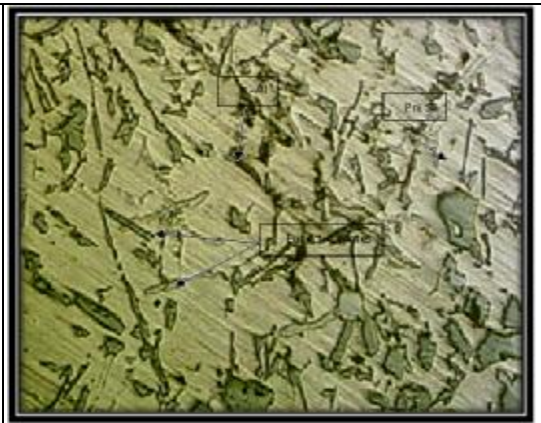**Figure (4):** The foam samples produced by one stage route foamed with deferent CaCO$_3$ percents and foaming temperatures (1,4,7**:**0.5%,700˚C ,750˚C,800˚C) , (2,5,8**:**1%,700˚C,750˚C, 800˚C) ,(3,6,9**:**1.5%,700˚C,750˚C,800˚C).

| **Figure(6):** Microstructure of foam precursor containing 1% CaCO$_3$ (X500). | **Figure(5):** Microstructure of as cast Al-12Si alloy(X250). |

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                              ┌──────────────────────────┐
                              │  Melting (2kg) of Al-12Si │
                              │    at 850°c in steel      │
                              │         crucible          │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │  Start agitation at       │
                              │  (1200)rpm in furnace     │
                              └────────────┬─────────────┘
                                           ▼
┌──────────────────────┐      ┌──────────────────────────┐
│ Mixing Al-12Si powder│      │  Continue agitation       │
│    &CaCo3 powder     │      │  inside the furnace till  │
└──────────┬───────────┘      │  temperature of crucible  │
           │                  └────────────┬─────────────┘
           ▼                               ▼
┌──────────────────────┐      ┌──────────────────────────┐
│ Heating the mixture  │─────▶│  Adding foaming agent     │
│      to 300°c        │      │  gradually with agitation │
└──────────────────────┘      │  at (1200) rpm till       │
                              │  temperature of crucible  │
                              │         fall to           │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │  Remove out the crucible  │
                              │  and pouring the melt in  │
                              │      a rectangular        │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │  Cooling the mould in air │
                              │  and remove the precursor │
                              │        from mould         │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │ Sectioning in a required  │
                              │         sizes             │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │ Insert the precursor      │
                              │ portion in backing die    │
                              │ and located the die in    │
                              │     backing furnace       │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │ Remove out the die ,      │
                              │ cooling by jet of air and │
                              │ remove out the sample     │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │ Sectioning to the required│
                              │ dimensions for testing    │
                              └────────────┬─────────────┘
                                           ▼
                              ┌──────────────────────────┐
                              │      Investigation        │
                              └──────────────────────────┘
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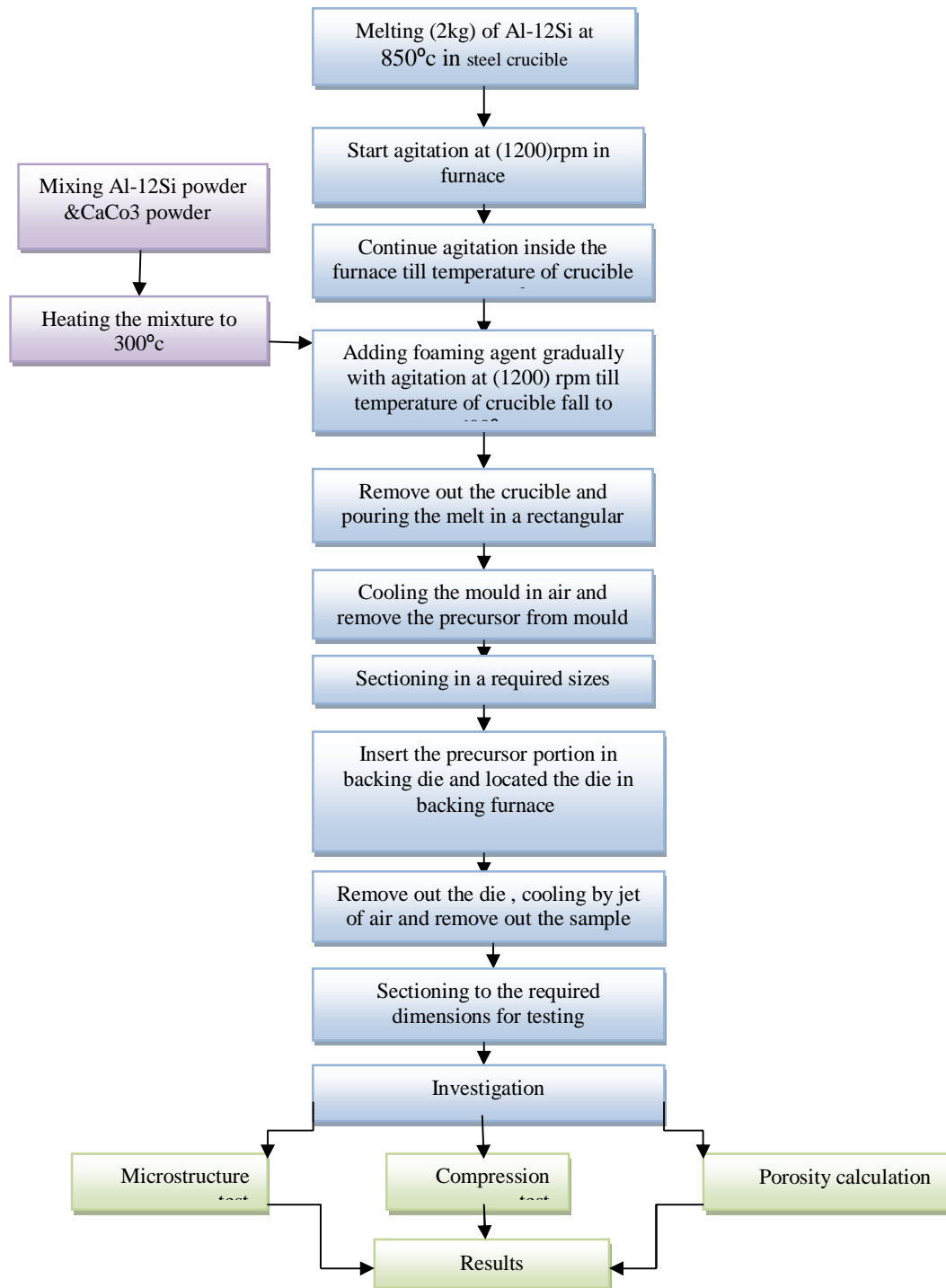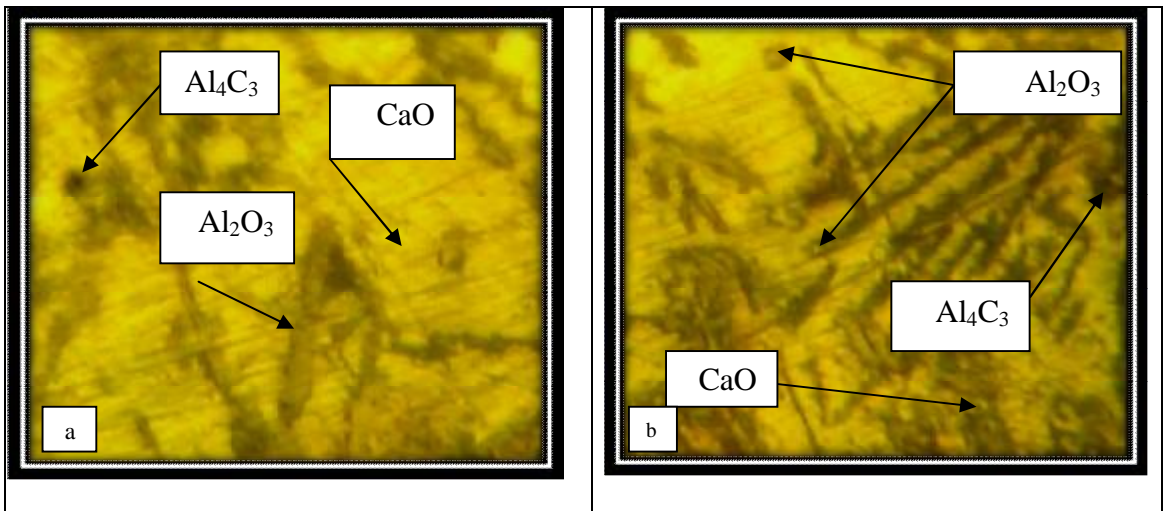| Microstructure test	Compression test	Porosity calculation

Results

Figure (3): The block diagram of foam fabrication using precursor technique.

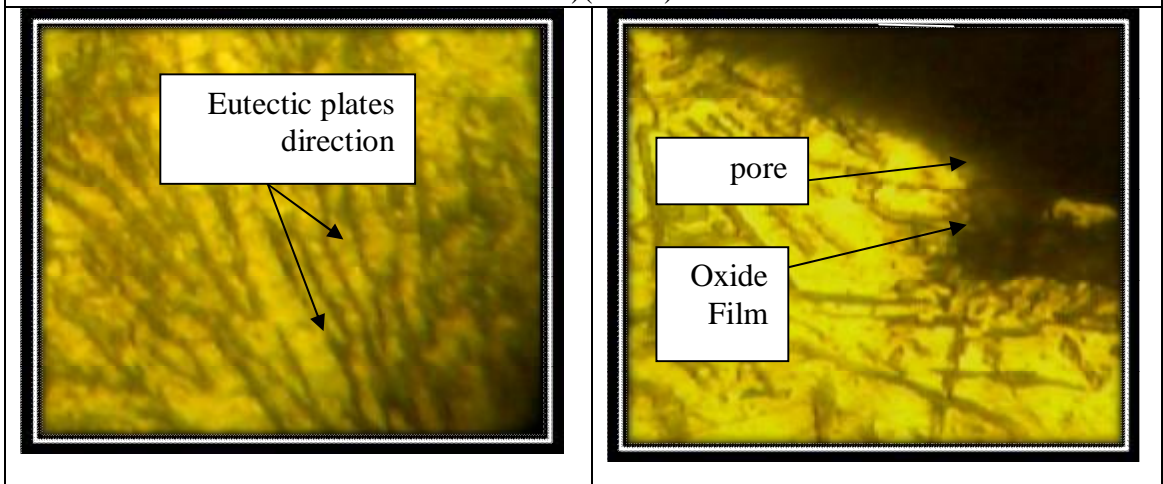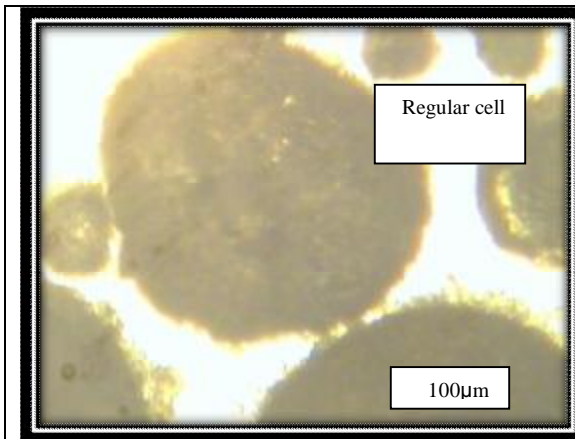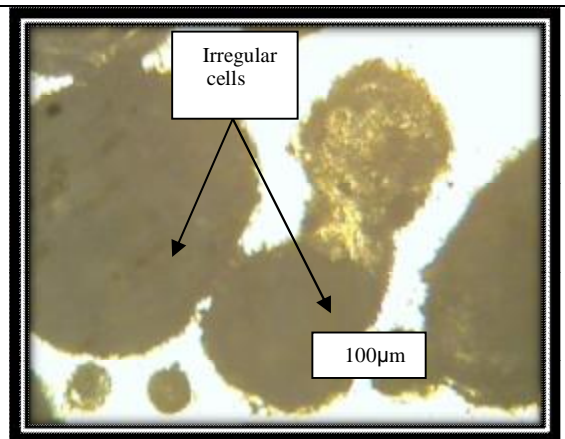**Figure(7):** Microstructure of metallic foamed with 1% $CaCO_3$ at (a: 700˚C , b: 750˚C)(X500).

Figure (8): Microstructure of metallic foamed with(1%)$CaCO_3$ at (800˚C) shows the region near the pore .

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Regular cell

100µm

Figure(9): The microstructure of metallic foamed with (1%)CaCO3 at(750˚C) with regular foam cells .

Irregular cells

100µm

Figure(10): The microstructure of metallic foamed with (1.5%)CaCO3 at(750˚C) with irregular foam cells .

Figure(11) Sample foamed for(5min) .

Figure(12): ample foamed for(15min). The mechanism of foam formation is a function of foaming time [4], but the metallic foams are unstable systems because their large surface area causes energy to be far from a minimum value [13] .
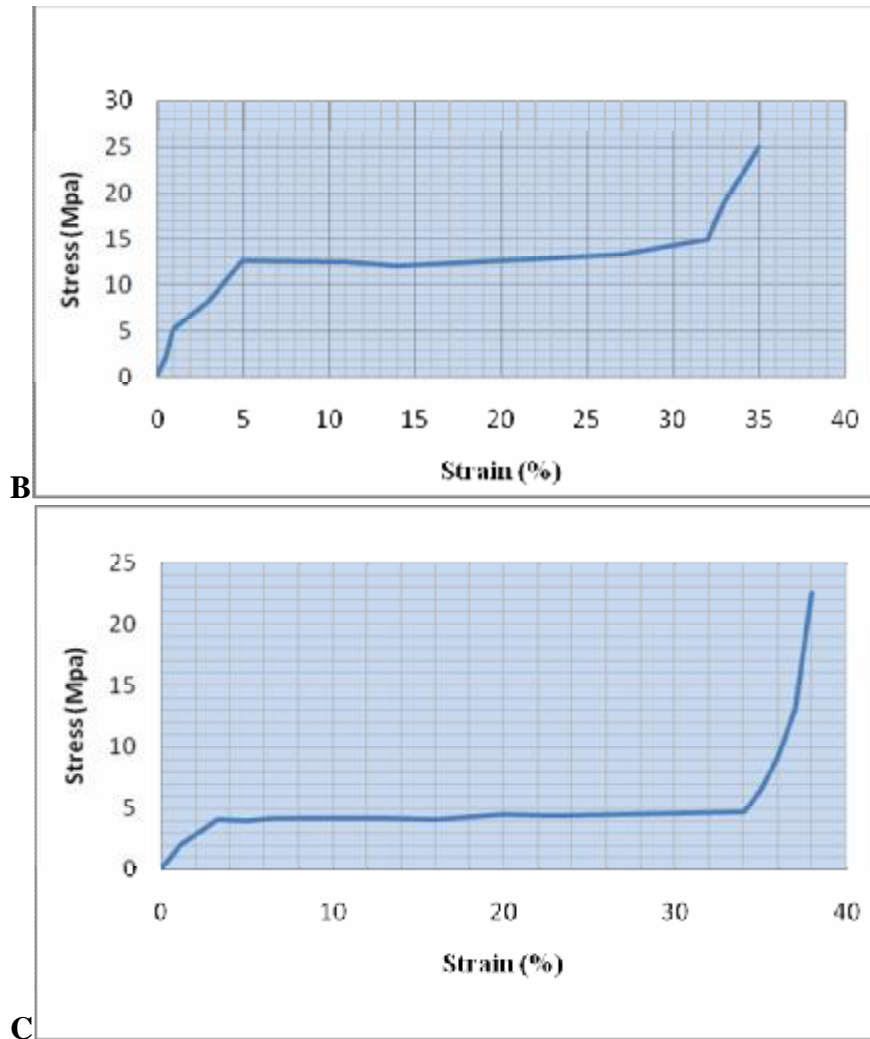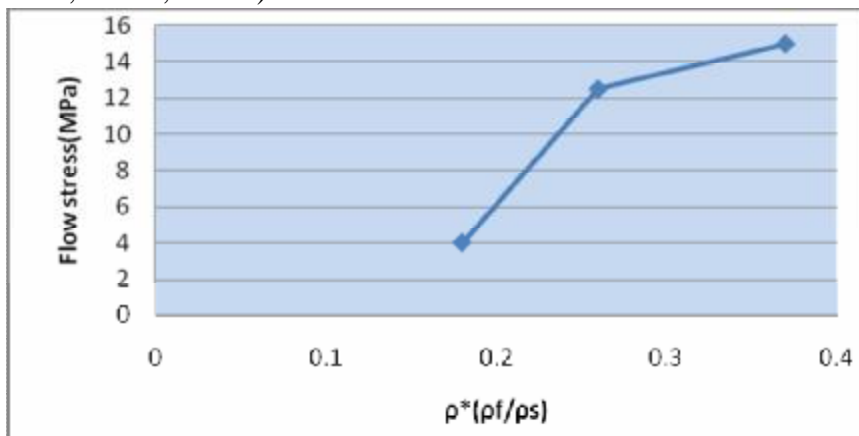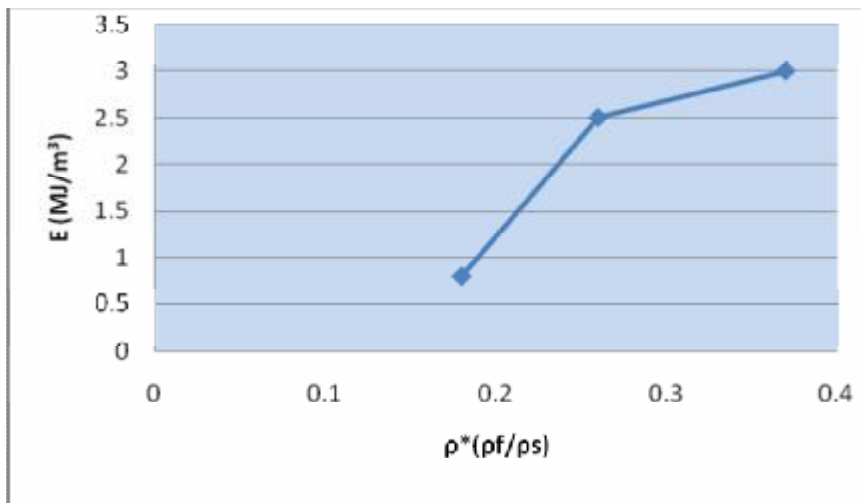
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Figure(13): Relationship between foaming temperature and porosity at different CaCO$_3$ percents.

A

B

C
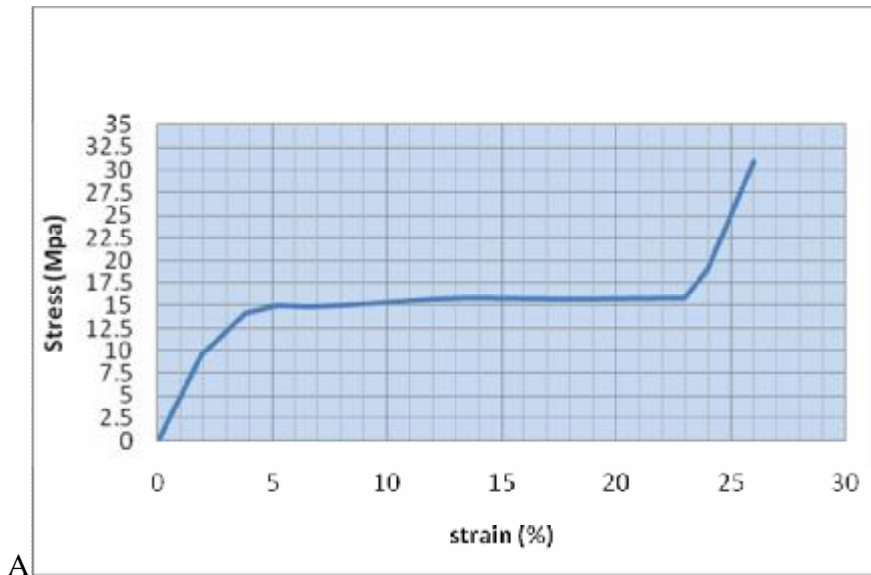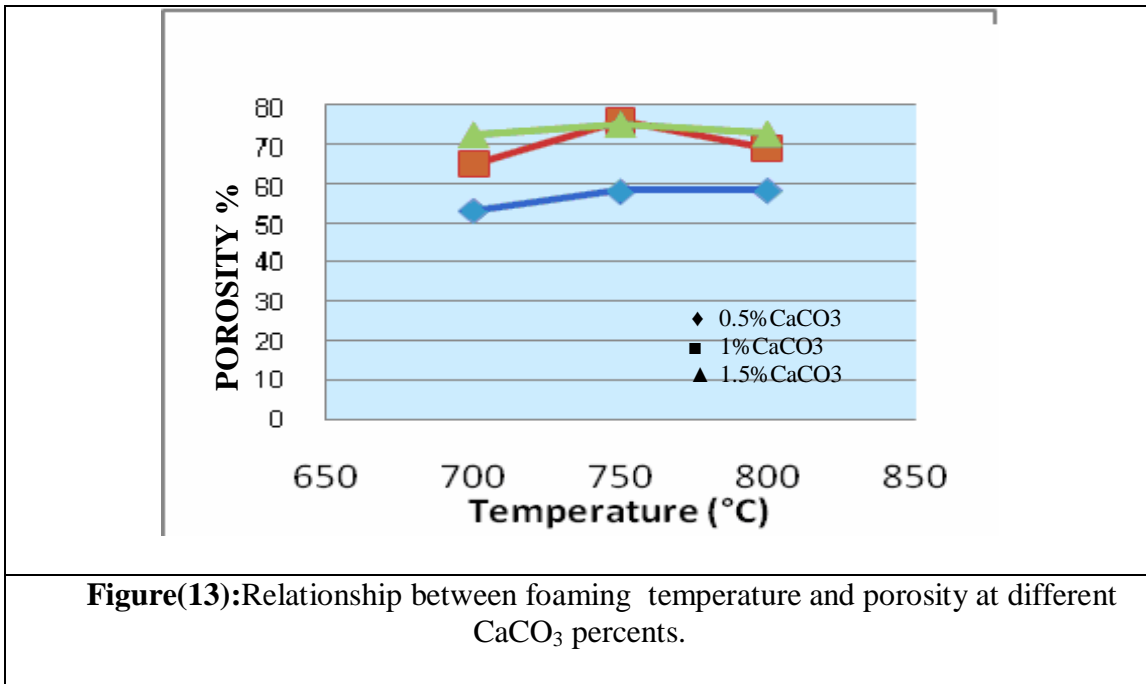
Figure (14): Stress strain curve for foam samples with relative density of (A: 0.37,B:0.26,C:0.18).

Figure(15): Relative Density(ρ^*) and Flow Stress Relationship.

Figure(16): Relative density and Energy Absorbed Relationship of metallic foam samples.