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THE EFFECT OF ADDING CALCINED ALUMINA ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ALUMINUM FOAM

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Abstract
In the last decades, Aluminum foam has attracted many researchers and manufacturers due to its unique properties which find a lot of applications, especially in a lightweight structure. The current work aims to study the effect of adding calcined alumina to the melting aluminum on the final mechanical and microstructure of produced aluminum foam. Calcium carbonate was used as a foaming agent. The study reveals that calcination times/temperature have a significant effect on the final foam porosity. with increasing calcination time from 30 min to 90 min the foam density improved from 2.27 to 1.27 g/cm^3, which consequently result in increase of samples porosity from 15.9 to 52.7%. also, the results from the compression test show that the variation of calcination time can be used to alter the energy absorption capacity of samples. Tested

samples achieved the highest energy absorption of 128 MPa at a calcination time of 90 min. Also, the study reveals that calcination temperature has a significant effect on the final foam porosity. with increasing calcination temperature from 400 to 800°C, the foam density improved from 2.18 to 1.09 g/cm^3, which consequently result in increase of samples porosity from 18.94 to 52.79%. Tested samples achieved the highest energy absorption of 325 MPa at a calcination temperature of 800°C. The increase in porosity is measured by ImageJ software, and the energy absorption test was carried out by a quasi-static compression test. These increases produced material can be used in applications needs high energy absorption like automotive industry.

Keywords: Aluminum foam, Calcined alumina, Lightweight materials, Blowing agent, Energy absorption

1 Introduction

Metal foams are cellular materials, which have many interesting mechanical and physical properties, such as high energy absorption during deformation, high stiffness, and very low specific weight. These outstanding properties make aluminum foam used in various applications such as railway, automotive, shipbuilding, architectural panels, and aircraft industry[1][2][3][4]. This broad use of aluminum foaming technique is always evolving due to the importance and increase of usage of this technology. Some studies improve properties by the change between various methods in manufacturing such as the metal vapor method, liquid metal, powdered metal, and metal ions method. All of the fabrication methods were described in studies[5][6][7][8][9]. The most straightforward technique to manufacture foams is to use a foaming agent with the addition of alumina to aluminum foam[10][11][12]. That showed good effects on the microstructure and mechanical properties of foams with the addition of Alumina. Results showed composite foam with 3 wt.% of Alumina nanoparticles had a more uniform microstructure and more channels than other samples. The Vickers hardness values of the composites foam increased with an increase in wt.% of alumina particles[11]. The effect of addition alumina was When 3 vol% Alumina is added, the cell shapes and sizes are not affected significantly. When the amount is increased to 5 vol%, cells become larger, but there is still a fine structure, and except a few numbers of cells, the cell size is homogeneous. The structure significantly changes

when an Alumina content increases to 10 vol%. The number of cells decrease, and cell sizes become larger. This is considered a weakness in the sample[10]. In the study of "Effects of calcination temperature on the pore size and wall crystalline structure of mesoporous alumina"[13], it studied the effect of temperature of alumina calcination on foam. Testing results indicated that the pore size was increased with increasing calcination temperature. And best alumina calcination temperatures between 550 and 1100 °C. So 800 °C was a suitable temperature for calcination alumina[13][14][15].All these studies made improvements in the properties of Aluminum foam by the addition of calcined alumina to foam[16][17][18][19].The most important of these are microstructure properties such as homogeneous pores, porosity percentage, and mechanical characteristics as energy absorption). Aim of this work is to investigate in the effect of adding calcined alumina at different alumina calcination times and different alumina calcination temperatures to improve properties of aluminum foam.

2 Experimental Procedure

2.1 Materials and Equipment

In this work the used Alumina and Aluminum obtained from the Egypt aluminum company from Qena, with a chemical composition as shown table 1. The used aluminum with purity 99.7%wt. as shown in table 2.

Table 1: Chemical composition of alumina

Alumina chemical composition	**SiO_2**	**Fe_2O_3**	**TiO_2**	**V_2O_5**	**%Na_2O**	**CaO**	**ZnO**	**MnO**	**P_2O_5**	**moisture at**	**bulk density**	**angle of respons e**
unit	%	%	%	%	%	%	%	%	%	%	gm/cm^3	degree
result	0.020	0.001	0.003	0.001	0.450	0.050	0.001	0.001	0.001	0.500	0.950	32.00

This work was used an electric furnace to melt the aluminum and calcine the alumina. The furnace uses a direct voltage source of 220V and reaches temperatures up to 1200 °C. And motor for stirring process with a maximum speed of 1500 rpm, this motor linked with mixer (rod 15 mm radius, length 800 mm, at its end two blades for good stirring). The

sample was melted in a crucible made from steel with dimensions of 90 mm diameter, and 100 mm high as shown in Figure 1.

Table 2: Chemical composition of Aluminum

	Si	Fe	cu	Mn	Mg	Cr	Zn	Ti	Al
Aluminum chemical composition	0.090	0.110	0.000	0.001	0.003	0.001	0.004	0.002	99.76

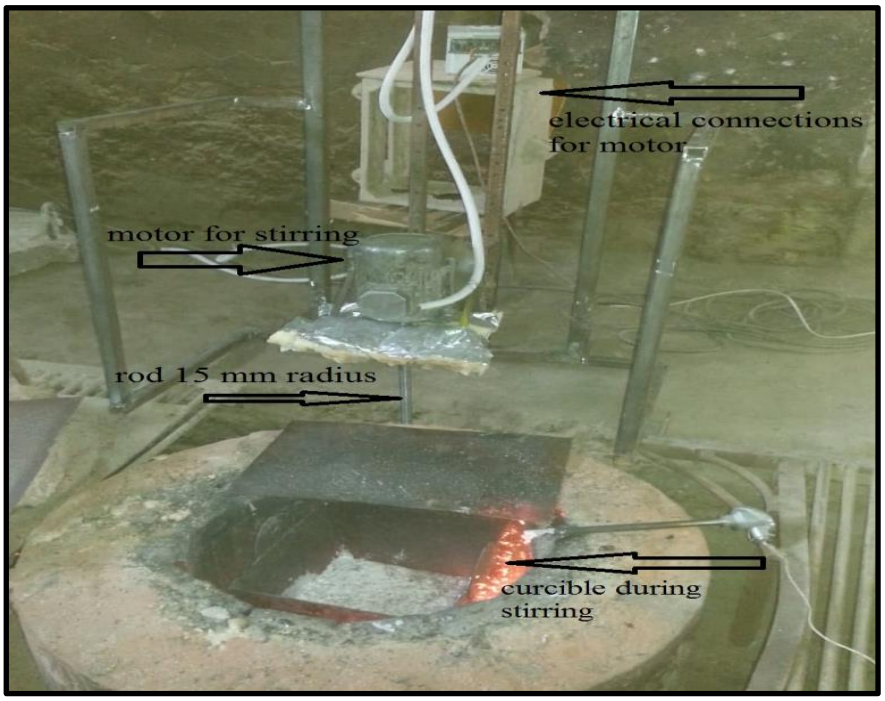

Figure 1 Set up for manufacturing process

ImageJ software program was used for analyzing the microstructure of Aluminum foam samples. ImageJ software was used in the current study to create density charts and line profile plots for produced samples. the number of pores and pore size diameter and total foam in area also were measured. The quasi-static compression test is done by a universal testing machine that uses a maximum load of 30 tons and data logger TDS150 (Copyright 2008 Tokyo Sokki Kenkyujo Co., Ltd.) to read results, test made at Assiut University as shown in Figure 2.

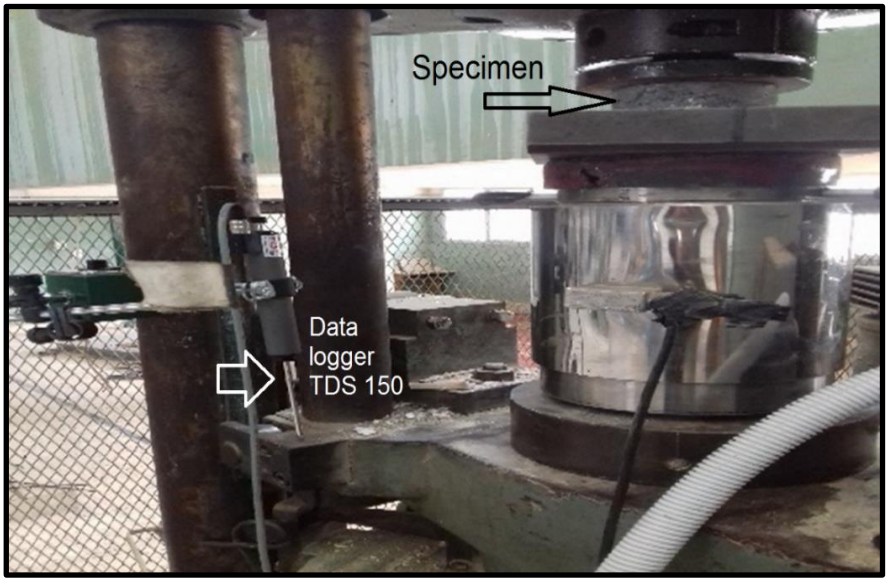

Figure 1. Aquasi-static test with universal testing machine& max load 30 ton

2.2 Foam Production:

2.2.1 preliminary experiments before calcination alumina effect

A sample weighing 500 g was left to melt inside the furnace at a temperature 700 °C. Then 3%wt. of alumina was added, because this added percentage of alumina produces more porous foam with a uniform microstructure as point out in the study[7]. Subsequently, 3%wt. of calcium carbonates was added to the mix, then the mixture was stirred. The stirring process causing activate the reaction, that creates the bubbles from the reaction: $CaCo_3 \rightarrow CaO + CO_2$ (gas) [6][20].

At first, the sample was not foamed due to the deposition of the molten aluminum because of the rapid cooling. This causes the sample to dry out before completing the stirring process which filled the pores with molten metal. This problem solved by a flame (oxy-acetylene welding torch) applied to the sample to preserve its molten state during stirring. Another problem that appeared is stirring speeds of 900 rpm are insufficient, so the speed was gradually increased to 1200 rpm, which resulted in a better foaming percentage as shown in Figure 3.

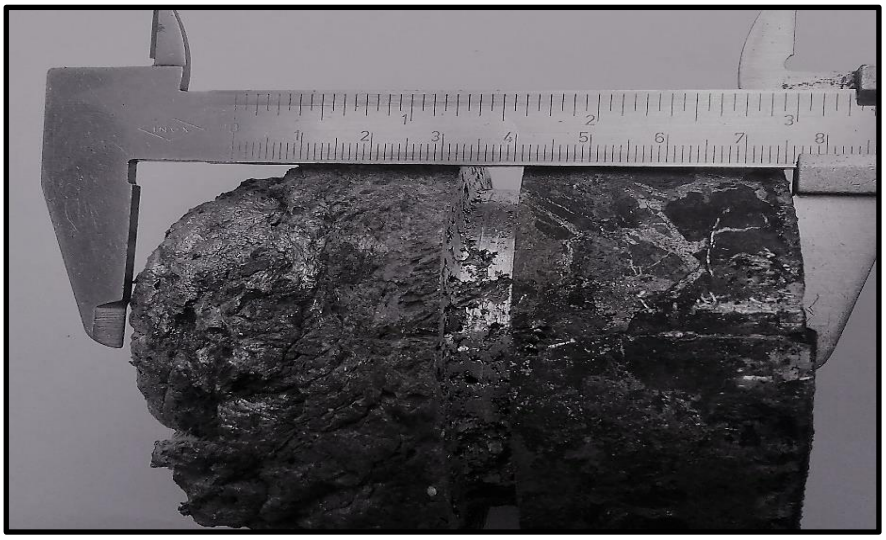

Figure 3. Sample before calcination alumina

2.2.2 *Experiment with Adding Calcined Alumina*

The calcination process is heating material to different temperatures in the absence or limitations of air or oxygen, or it is a thermal treatment process to remove water or CO_2 from hydrated oxides or carbonates As shown in Figure 4. For example:

$AL_2O_3.\ 2H_2O \rightarrow Al_2O_3 + 2H_2O$ (equation of alumina calcination)[18].

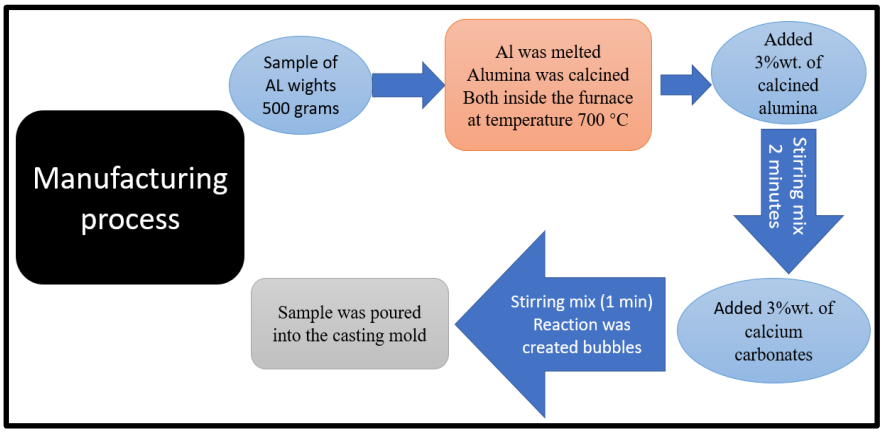

Figure 4. Manufacturing process of aluminum foam

Sample weighing 500 g, was manufactured by the same method as the previous sample. But the added percentage (3 wt.% alumina) was

calcined inside the furnace at temperature 800 °C, and time 1 hour. Then alumina and aluminum are poured together in the crucible and stirred for 2 minutes. Calcium carbonates was mixed in the melt and stirred for an extra 1 minute. Results shown low density, more pores as shown in Fig.5. That referred to high foaming percentage than the samples before using calcination alumina.

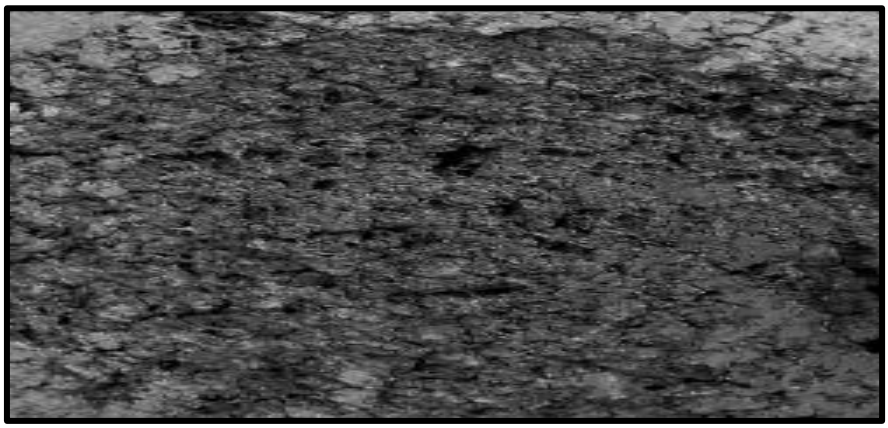

Figure 5. Scanned image of produced foam with adding calcined alumina

2.2.2 samples preparation and methodology:

The studied variables were including a variation of calcination time (30, 60, and 90 min). the temperature of the calcination process was (400, 600, and 800 C^{o}). The manufacturing samples were cut by a lathe machine to be ready for testing at dimensions (90 mm diam, high =33mm). The sample was prepared for tests as shown in Figure 6.

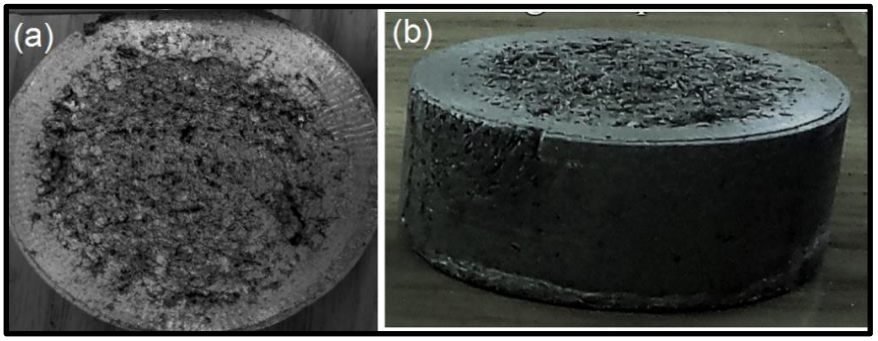

Figure 6. Foam sample prepared for tests (a) top view (b)side view

3 Results And Discussion

3.1 Calculation of Density and Porosity for the Six Samples

The porosity of foam (P_f) can be determined from the following equation:

$$P_f = | 1 - (\rho_{foam}/\rho_{al}) | *100\% \quad [8]. \tag{1}$$

where, ρ_{al} = aluminum density (2.7 g/cm^3), ρ_{foam} = foam density (g/cm^3) Results showed that with the increase of time and temperature of the adding calcined alumina, a reaction responsible for creating bubbles activated gradually. This activation of reaction increases the number of pores, average pore size, thus increase the total foaming area. This change in parameters decreases the density, increases sample porosity[13][14][21]. As shown in table (3).

Table 3: Calculation of density & porosity for six samples:

	variables		Samples after cutting. radius=4.2 cm, high =3.3 cm		Porosity percentage
	temperature	time	Mass Per g	Density Per g/cm^3	porosity %
1	400°c	30 min	415	2.27	15.91
2	400°c	60 min	400	2.18	18.94
3	400°c	90 min	233	1.27	52.78
4	400°c	60 min	400	2.18	18.94
5	600°c	60 min	208	1.13	57.85
6	800°c	60 min	200	1.09	59.47

3.2 Measuring porosity for samples by using ImageJ software

3.2.1 Samples when adding calcined alumina with variable time and constant temperature:

When adding calcined alumina at (400°C & 30 min) to melting aluminum, the results of measuring porosity by ImageJ and SEM images showed a decrease in the number of pores. Produced non-uniform microstructure foam, and size of pores decreased. With increasing time for calcined alumina to 60 min, 400 °C, the number of pores, and pore average size gradually increased. Total foaming area better than the first sample, due to increasing time of calcination alumina to 60 min. When adding alumina calcined at 400 °C & 90 min, the number of pores

increased, total farming area and average pore size increased than the first and second samples. Sample as shown in (table 4) and Figure 7. Results proved that when alumina calcination time increases, this leads to a good reaction that increases the number of pores and total foam area. This increases the porosity of samples[13][14][21].

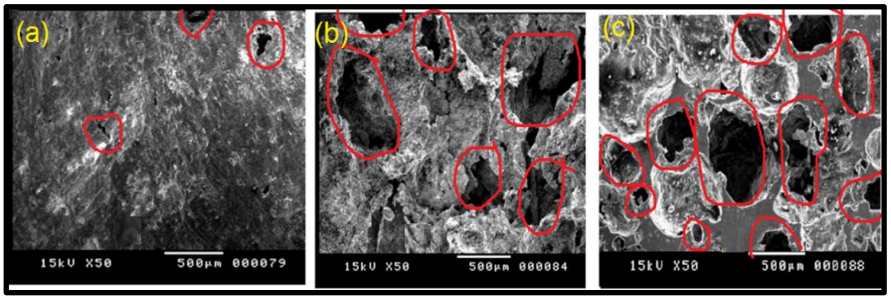

Figure 7. SEM images of produced foam when adding calcined alumina at (a) 400°C & 30 min (b) 400°C & 60 min (c) 400°C & 90 min

Table 4 : Calculations of no of pores, pore size and by using ImageJ.

Sample variable time and constant temperature 400 °C	No of pores	Total Area mm^2	Average Size diameter mm
30 min	1335	22.560	0.017
60 min	2608	81.090	0.031
90 min	5877	536.350	0.091

3.2.2 Samples when adding calcined alumina with variable temperature:

At first, added the calcined alumina at a temperature of (400°C & 60 min) to the sample. This sample was showed a lower number of pores 2608 and non-uniform distribution of pores. Then increase of the temperature of alumina Calcination to 600 °C at a constant time of 60 min, which showed the number of pores and average pore size improved gradually than the previous sample, due to the increase of temperature of calcined alumina to 600 °C. In case of increased temperature of calcined alumina to 800°C and constant time 60 min, the number of pores increased to superior value 9569, and pore size decreased. That is an indication of a uniform distribution of pores of microstructure and a high percentage of foamability that reaches 59.47 %. Results are represented in table (5) and Figure 8. Obviously, with increasing calcination temperature the reaction who created bubbles activated. that created more pores and pore diameter increases and the pore size distribution comes wider thus, cause increasing porosity of the foam[13][14].

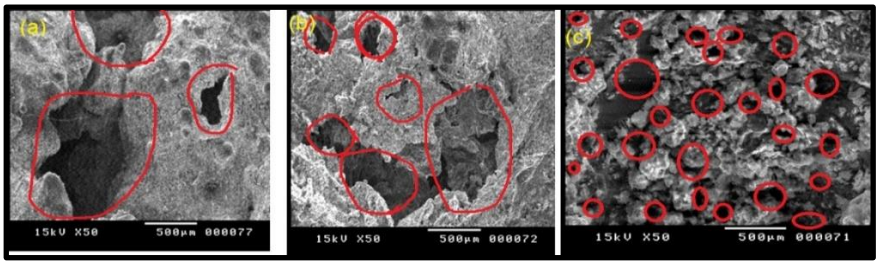

Figure 8. SEM images of produced foam when adding calcined alumina at (a) 400°C & 60min (b) 600°C & 60 min (c) 800°C & 60 min

Table 5: Calculations of number of pores, pore size and total foaming area by using ImageJ software.

Sample variable temp and 60 min	**No of pores**	**Total Area** mm^2	**Average Size mm**
400°C	2608	81.099	0.031
600°C	5237	907.046	0.173
800°C	9569	526.706	0.055

3.3 Compression Test

Compression tests were carried out at room temperature by a universal testing machine that uses a maximum load of 30 tons and data logger TDS150 using a 100 kN load cell and a crosshead speed of 1.0mm/min. Testing machine was programmed for the determination of energy absorption by the equations[21].

$F = M_{(\text{from 1 to 30 ton})} * G$ (2) The plateau stress $\sigma = F/A_{(\pi * 0.09^2)}$ (3)

Strain $\varepsilon = \Delta L_{\text{elongation}}/L_{\text{length}}$ (4) Energy absorption $E_V = \sigma * \varepsilon$ (5)

Where energy absorption E_V can be evaluated for a compressive strain of 0.0 to 0.5 and (σ) is the compressive stress and ($\boldsymbol{\varepsilon}$) is the compressive strain. Therefore, plateau stress is closely related to the amount of energy absorption E_V. If the plateau stress of porous aluminum can be estimated by a simple process. energy absorptivity was evaluated comparatively easily from the study [9][21].Test as shown in Figure 9.

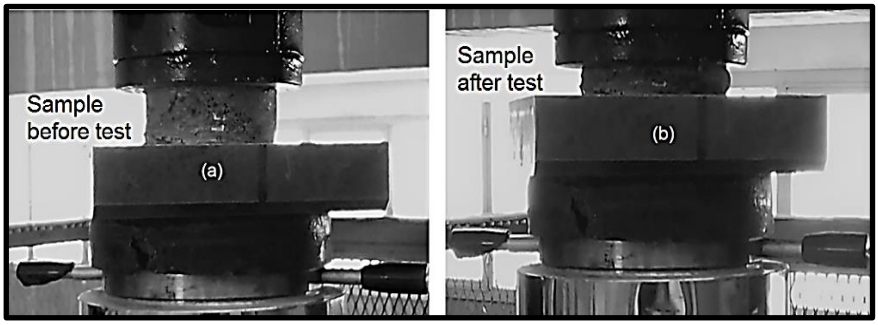

Figure 9. A quasi-static compression test for samples
(a) original specimen before test - (b) specimen after test

3.3.1 Stress-strain curves when adding alumina calcined at a variable time :

Compressed sample let the material crystals or atoms fill the pores, which is an indicator of the compressibility of the material and the energy absorption capacity of the manufactured material. The results proved that when the calcination time of the alumina is increased and then added to the sample, this results in a good reaction resulting in more pores and increases the diameter of pores. The increase in the porosity helps more in the sample's compressibility, which improved the energy absorption[13][14][21]. When alumina calcination time increases to 1 hour at 400°C. Energy absorption reaches the highest value up to 85 MPa. Results are represented in the stress-strain curve in Figure 10.

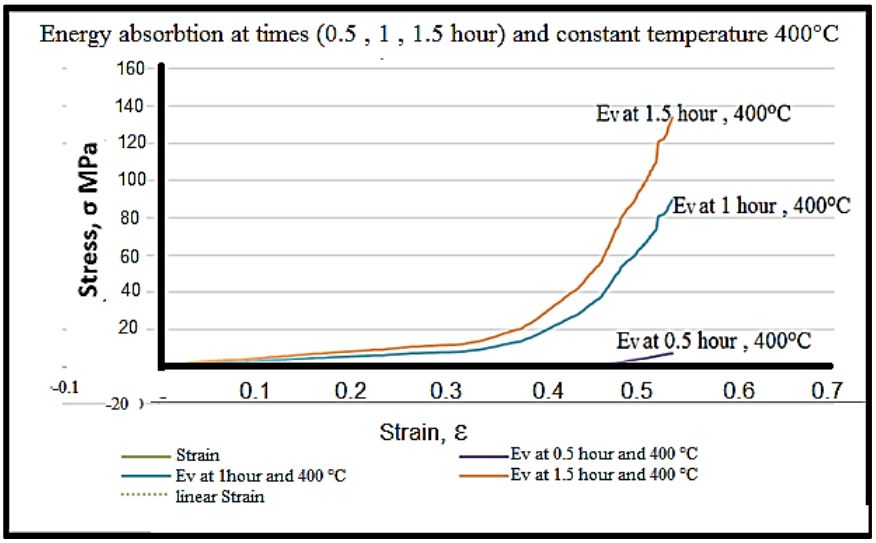

Figure 10. Stress-strain curves when adding alumina calcined at a variable time (0.5hour, 1 hour, 1.5 hour) and constant temperature 400 °C

3.3.2 Stress-strain curves when adding alumina calcined at variable temperature:

The increase in temperatures of the adding calcined alumina affected on energy absorption clearly. So, the value of E_V at 800°C reaches a maximum of up to 325 MPa. The sample was compressed from 34mm to 18mm. The test proved that with increasing alumina calcination temperature from 400°C to 600°C energy absorption increased, and at 800 °C yielded the best.

Obviously with increasing calcination temperature the pore diameter increases, and the pore-size distribution be- comes broader Thus, when the porosity increases, compression of the sample is easier to occur due to the increased voids within the sample, which also improves the energy absorption property. That represented in the stress-strain curve in Figure 11.

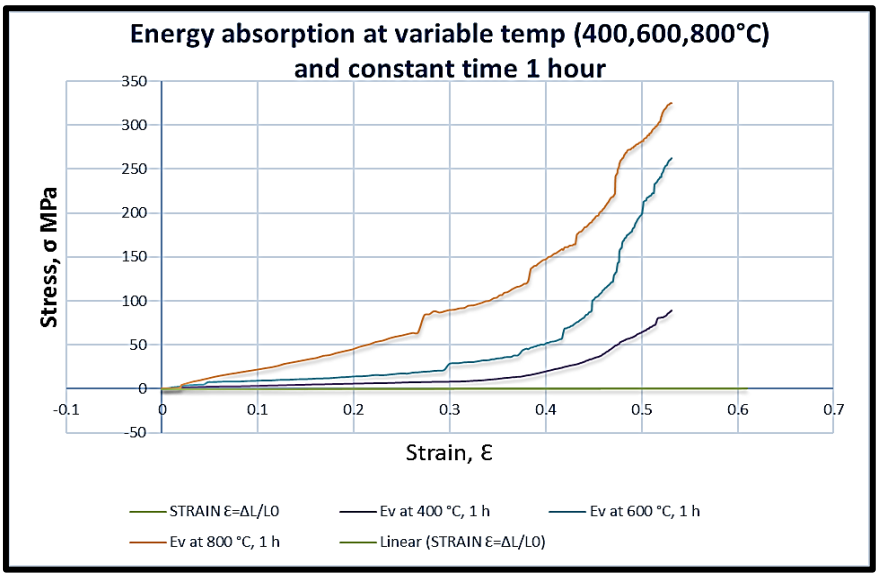

Figure 11. Stress strain curves when adding alumina calcined at 400 °C, 600°C, 800°C and constant time 1 hour

Values in the curve proved the significant differences between the results, which indicate a significant improvement in the ability of samples to absorb energy with increasing calcination time for alumina and the reason is the reaction activity observed when increasing calcination time which leads to an increase in the number of bubbles resulting, causing increased porosity Increasing porosity gains the

samples compressibility and increasing the number of pores induces the metal atoms to slip when pressed, which shows the property of absorbing energy. As shown in Figure12. Values of Fig.13 showed the highest improvement in the values of energy absorption between different calcination temperatures. When temperature be 800 °c graph reached to highest values as compared with the average graph**.**

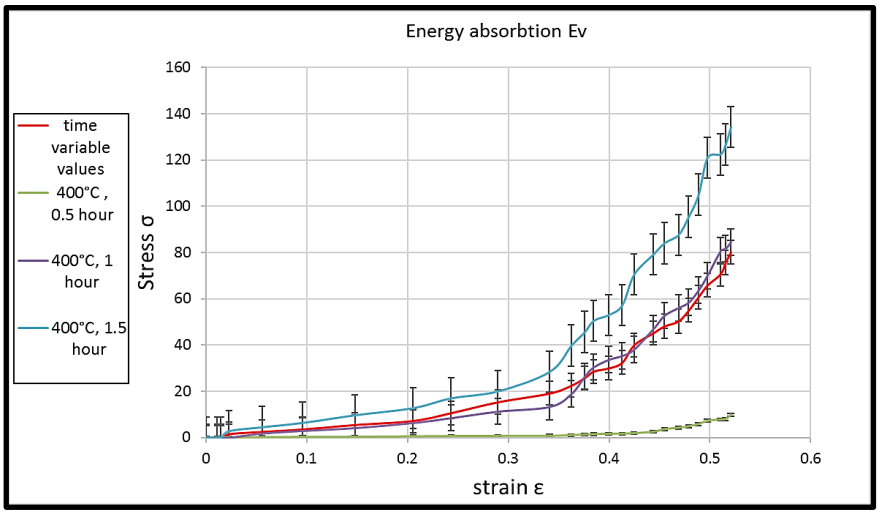

Figure 12. Energy absorption variation when calcination time variable (30,60,90 min)

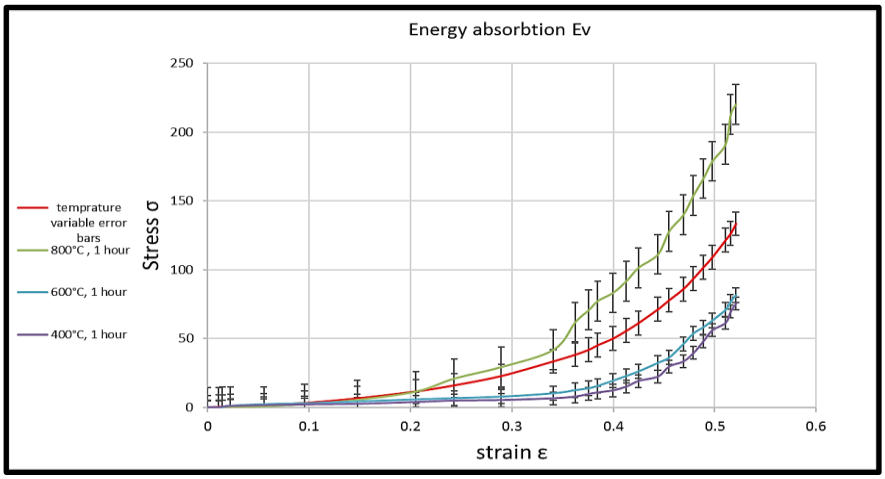

Figure 13. Energy absorption variation when calcination temperature variable (400,600,80 °c)

4 Conclusions

The following conclusions can be drawn from this work: Adding calcined alumina can be efficient for producing aluminum foam at higher calcination temperatures than 600°C and time 1hour.

- When the time of the calcination alumina increased from 30 to 90 min and temperature 400 °C. The foam density improved from 2.27 to 1.27 g/cm3, which consequently result in an increase of samples' porosity from 15.9 to 52.7%. also, the results from the compression test show that the variation of calcination time can be used to alter the energy absorption of samples.
- The temperature of alumina calcination increased up to 800°c. the foam density improved from 2.18 to 1.09 g/cm3, which consequently result in an increase of samples' porosity from 18.94 to 52.79%. Tested samples achieved the highest energy absorption of 325 MPa. That refers to improvement in foam production with an increase of temperature of the calcined alumina addition. Tested samples achieved the highest energy absorption of 128 MPa at a calcination time of 90 min.

These improvements in sample porosity and energy absorption due to adding of calcined alumina proved the aim of this work.

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دراسة تأثير إضافة الألومينا المكلسة على البنية المجهرية والخواص الميكانيكية لرغوة الألومنيوم

الملخص

في العقود الأخيرة ، اجتذبت رغوة الألومنيوم العديد من الباحثين والمصنعين بسبب خصائصها الفريدة التي تجد الكثير من التطبيقات ، وخاصة بسبب هيكلها الخفيف الوزن. . يهدف العمل الحالي إلى دراسة تأثير إضافة الألومينا المكلسة إلى الألومنيوم الذائب على البنية الميكانيكية والمجهرية النهائية لرغوة الألومنيوم المنتجة. تم استخدام كربونات الكالسيوم كعامل رغوة. تكشف الدراسة أن أوقات التكليس / درجة الحرارة لها تأثير كبير على مسامية الرغوة النهائية. مع زيادة وقت التكليس من ٣٠ دقيقة إلى ٩٠ دقيقة ، تحسنت كثافة الرغوة من ٢,٢٧ إلى ١,٢٧ جم/سم ٣ ، مما يؤدي بالتالي إلى زيادة مسامية العينات من ١٥,٩ إلى ٥٢,٧٪. أيضا ، تظهر نتائج اختبار الضغط أن اختلاف وقت التكليس يمكن استخدامه لتغيير قدرة امتصاص الطاقة للعينات. حققت العينات المختبرة أعلى امتصاص للطاقة ١٢٨ ميجا باسكال في وقت التكليس ٩٠ دقيقة. أيضا ، تكشف الدراسة أن درجة حرارة التكليس لها تأثير كبير على مسامية الرغوة النهائية. مع زيادة درجة حرارة التكليس من ٤٠٠ إلى ٨٠٠ درجة مئوية ، تحسنت كثافة الرغوة من ٢,١٨ إلى ١,٠٩ جم / سم ٣ ، مما يؤدي بالتالي إلى زيادة مسامية العينات من ١٨,٩٤ إلى ٥٢,٧٩٪. حققت العينات المختبرة أعلى امتصاص للطاقة يبلغ ٣٢٥ ميجا باسكال عند درجة حرارة تكليس تبلغ ٨٠٠ درجة مئوية. يتم قياس الزيادة في المسامية بواسطة برنامج ImageJ ، وتم إجراء اختبار امتصاص الطاقة بواسطة اختبار ضغط شبه ثابت. أثبتت هذه الاختبارات بوضوح أن التحسن في خصائص المسامية وامتصاص الطاقة بسبب زيادة الوقت ودرجة حرارة تكليس الألومينا. التي أشارت إلى قدرة امتصاص الطاقة العالية للرغوة المصنعة ، ويمكن استخدامها في التطبيقات التي تحتاج إلى امتصاص طاقة عالية مثل صناعة السيارات.