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Improvement of the mechanical and thermal characteristics of open cell aluminum foams by the electrodeposition of Cu

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ABSTRACT

Recently aluminum foaming has been of much interest due to its characteristics properties of light weight structure. Metallic foams are highly porous materials which present complex structure of three-dimensional open cells. This aspect causes strong limitations in mass transport due to electro-deposition technology. In this work, the electro-deposition of copper on aluminum open-cell foams substrates was developed, in order to enhance the thermal and mechanical properties of these cellular materials. The mechanical and thermal characterization of the produced samples was lead through compression and conductivity tests. On the basis of the experimental results, analytical models are proposed to predict the quantity and the quality characteristics of the coating.

1. Introduction

In recent years, cellular materials were developed and used in almost every fields of engineering because of their unique structural and functional properties [e 1–3]. The excellent combination of good mechanical properties (mostly strength and stiffness) and low weight is the prime advantage for such class of materials. These features are function of several parameters as base material, mean pore size and distribution and density [e 4–8]. Metal foams with open cell structure have attractive functional properties. The particular inner structure of these foams permits the application of these materials in many technologies domains like heat exchangers [9–11] air and water cleaning [1], orthopedic applications [12]. Therefore, it is easy to understand why cellular materials are employed in a wide range of applications.

Metal foams are produced with several common used methods like powder compact melting method [13,14], dissolution and sintering process [13,15], replication process [1,13], melt-gas injection, melt-foaming agent, casting, etc. [13]. The parameters of each process influence the properties of the material, thus there are vary examples of optimization of process in literature [1,12]. However, the cost linked with the production of cellular materials is still too high, due to process complexity and hazard, although all the efforts to develop a cheaper procedure [1,6].

In this work, the electro-deposition of copper on aluminum open-cell foams substrates was developed, in order to enhance the thermal and mechanical properties of these cellular materials and, at the same time, to reduce production costs. In fact, the costs related to the direct production of copper foams are more consistent than those related the electro-deposition one in which the raw material cost three times less.

Coppering is often used also as intermediate step before nickel or chromium deposition [16]; it can be carried out using different kinds of bath and, among them, an acid copper sulfate solution was chosen because characterized by easy preparation of the reagents, simple effluent treatment, economy, high operative density current and homogeneous deposits. Again, in literature there are several researches about the effects of coatings on mechanical and thermal features of metallic bulk substrates, but only a few focus on foams substrates [17,18], which is more complex because the intricate inner structure of cellular materials causes strong limitation in mass transport. In this work one model of electrodeposition of copper and two models of optimization are proposed and validated in order to predict the quantity and the quality of the cupric coating. All the theoretical methods previously formulated were confirmed. In particular the influence of the preparation of the surface of aluminum was studied as first step of all the process. Generally, surface preparation is an essential part of electro-deposition coating mechanical polishing and electric adaptation, so as to remove oxides and organic layer. For this purpose, electric polishing and chemical agents like distilled water, acetone, and Trichlorethylene are currently used. The preparation studied in this work was carried out by means of sandblasting. This treatment provides to the elimination of the oxide layer that covers the surface of aluminum foams and which is the cause of irregular depositions. The results show a great improvement of

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the electro-deposition process, with a significant reduction of the time process, and the achievement of a continuous layer of coating on the foam substrate. The used values of current vary from 1.65 A to 3 A, whereas time is $7.2 * 10^3$ s at maximum (the optimum falls long before). In literature are present works in wich superficial preparation is not exploited [19].

The mechanical and thermal characterization of the produced samples was leaded through compression and conductivity tests. The stress–strain curves show the influence of the coating and the density on the mechanical behavior of the foams. Furthermore, it is possible to define three regions in these graphs: the elastic zone, the plateau zone and a break zone, in perfect agreement with literature results [20]. Heat exchange properties of the samples were studied by means of an experimental device, built ad hoc to suit the particular characteristic of this material. Also in this case, the combination with copper promotes an improvement of the thermal exchange properties, as expected. On each sample an image analysis through optical and scanning electron microscope (SEM) was leaded.

2. Experimental procedure

Aluminum foams were chosen as substrate for the electrodeposition because they are lightweight, easy to produce, widely used and cheap, just to underline their most important features. In particular an aluminium alloy 6101 T6, produced by "ERG Material", was chosen. All the samples were obtained from the same block, which was produced through the replication of polymeric pattern method. With this process, a polymeric open-cell foam of convenient porosity is covered with a slurry (ceramic powder and water) which, after a heat treatment, dries and hardens while the polymer melts leaving a void that can be filled with a metal, in this case aluminum. The specimens were manufactured with the same parallelepiped shape ($20 \times 20 \times 50$ mm), however their density covers a quite large range of values, from 150 to 250 kg/m^3, which is much lower than the specific weight of the base material (2700 kg/m^3), with an average pore size of 10 ppm ($2.237 * 10^3$ μm).

An acid solution based on copper sulfate was chosen as electrolytic bath, due to its elevated anode dissolution efficiency that is nearly close to 100%. Its composition was studied and compared with those found in literature and consisted of 1.25 M $CuSO_4$, 0.61 M H_2SO_4 and Cl^- 50 ppm. The presence of chloride is helpful in the polarization of anode and in the modification of the characteristic of the coating, whereas $CuSO_4$ and H_2SO_4 are obviously indispensable for the correct development of the process. The bath was kept in agitation with a magnetic agitator, located inside the electrolytic cell. The influence of the magnitude of the agitation had been studied in a previous phase of the experimentation, and the results leaded to choose 3.5 rpm for this parameter.

The consumable copper anode has been designed and manufactured in such a way that it can be adapted to the shape of the foam, obtaining the best possible results. The shape is that of a hollow tubular, composed by 4 faces screwed to support structure, whose dimensions are $50 \times 30 \times 5$ mm. Of course, all the assembled (including screws and nuts) is made of copper, so it is consumable: the sides are designed to be changeable so they can be replaced, once eroded, with new spare blades. The anode was thus also designed to make easily accessible that part of the metal foam which is always above the free surface of the solution, because connected to the terminal of the current generator. It was established, in fact, for the repeatability of the tests, that of 50 mm in height only 40 are those actually immersed in the bath and therefore subject to electrodeposition.

The surface preparation was carried out using a sandblasting machine, which exploits tiny glass spheres, at the pressure of 3 bar. This treatment eliminates the oxides that normally cover the surface of aluminum and that cause an imperfect and irregular coating. Since this undesired oxide layer regenerates itself spontaneously with time, the electrodeposition has to be carried out immediately after the sandblast. Moreover, was concluded that this method does not affect the mechanical properties of the foams, as showed by compression tests.

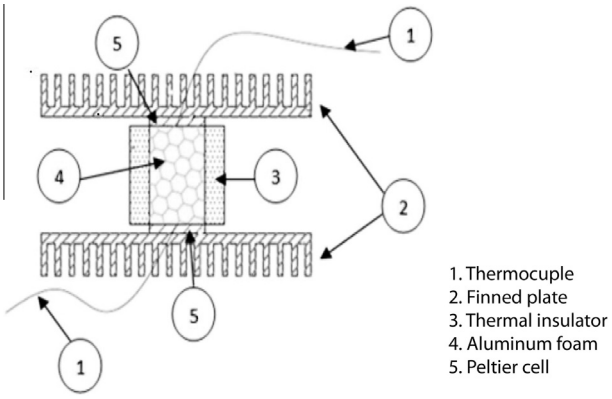

Fig. 1. The experimental device.

These test were leaded on each sample in a compression machine, setting a constant deformation rate of 5 mm/min and a 10 kN load cell. Before the test, the height of the specimens was set at 36 mm, in order to eliminate the possible dimensional differences and, at the same time, to remove that part of the foams that was not involved in the electrodeposition.

Conductivity tests were run, instead, in a particular experimental device. As showed in the Fig. 1, the foam is contained between two Peltier cells, which are electrically connected in series and can impose an established thermal flow through the foam itself. Since the topping cell warms and the bottoming one chills the foams, the resulting heat flow moves longitudinally with respect to the sample, whereas any possible deviation is prevented by the presence of an insulator material that surrounds the specimen. Moreover, two finned plates were integrated the extremity of the device in order to disperse the heat generated by the Peltier cell, on the faces not in contact with the specimen. Two thermocouples (tipe J) were welded to the bases of the foam, so as to monitor the temperature in stationary regime. Knowing the heat flow, the thickness of the foam, the gap of temperature between the bases and the amplitude of the exchange surface, it should be possible to calculate the thermal conductivity of the foams. However, since this surface is punctual, it was impossible to quantify its entity, although all the efforts accomplished in that sense. Furthermore, to prevent that the measurements were influenced by the presence of air within the completely chaotic structure of the foams, the comparisons about any improvement in thermal conductivity were carried out only on the same sample, before and after the electrodeposition process, and not between different specimens.

Finally, the standard of the achieved coating was tested by optical analysis leaded exploiting optical microscope and SEM. Before SEM analysis, the samples were conveniently polished in order to expose the area of contact between coating and substrate.

3. Theoretical models

The theoretical model exploited in this work has already been used and validated in a previous research. *P%* was chosen as the principal parameter for assessing the standard of the electrodeposition, and it is calculated as

$$P\% = ((P_2)_i - (P_1)_i)/(P_1)_i \quad (1)$$

where P indicates the mass of the samples before(subscript 1) and after (subscript 2) electrodeposition, whereas the subscript i indicates that these quantities are related only to the portion of foam immersed (thus only to the one that actually participates to the deposition). Therefore, $P\%$ indicates the amount of copper deposited compared to the initial immersed mass of the specimen. Developing the above expression, it can be written:

$$P\% = (MM_{Cu} * n_{Cu})/r * V_i \tag{2}$$

where MM_{Cu} is the molar mass of copper and n_{Cu} is the number of moles of copper electrodeposited. If it is considered the reduction half-reaction that occurs at the cathode:

$$Cu^{2+} + 2e^-, \rightarrow, Cu \tag{3}$$

it is noticed that, for every mole of copper ion that is deposited, two moles of electrons are required, thus:

$$P\% = MM_{Cu} * n_e/r * V_i * 2 \tag{4}$$

Calling e the electric charge of an electron, t the time(in minutes), i the electric current ad N_a the Avogadro's number, it is possible to rearrange the previous expression as:

$$\begin{aligned} P\% &= (MM_{Cu} * n_e/r * V_i * 2) * (N_a * i * t/N_a * i * t) \\ &= (MM_{Cu} * 60/N_a * 2e) * (i * t/\rho * Vi) \end{aligned} \tag{5}$$

It is noted that the first part of the second member of this equation is constant and assumes value $1.978 * 10^{-5}$ kg/A min.

This formulation expresses the relation that exists between $P\%$ and the parameters of electrodeposition: in particular, $P\%$ is directly proportional to the current and the time, and inversely proportional to the mass of the sample. Analyzing this expression, it is found that the numerator is a rearrangement of the Faraday law, whereas in the denominator there is the mass of the substrate. In particular, it has to be aware that ρ is not constant, as already said previously, therefore it has to be calculated for each samples: missing this passage could lead to rough errors, and it could cause an unpredictability of $P\%$. The possibility to foresee the trend of this parameters has been fundamental in this research, because the test scheduling was based on it. Thermal conductivity (λ) was established by the expression:

$$P = (\lambda/\delta) * S * \Delta T \tag{6}$$

where P is the thermal power exchanged, δ is the distance between the thermocouples, S is the exchange surface and ΔT is the gap of temperature between the bases. As it is possible to notice, the presence of air, and thus the presence of a mechanism of convective conduction, does not appear in the formula above. In other words, the system composed by aluminum and air is considered as a bulk material. However, as already said, the punctual nature of the surfaces makes impossible to determinate their extension, therefore only the relative improvement was calculated. In fact, considering constant S, for the same sample also after electrodeposition, it can be written that:

$$\lambda'/\lambda = [(P * \delta')/\Delta T'] * [\Delta T/(P * \delta)] \tag{7}$$

Making comparison on the same specimen allows us to ignore the contribute of air within the foam in heat exchange, which of course is almost the same before and after the electrodeposition. Moreover, it is noticed that sometimes $\delta' \neq \delta$ in the conductivity test could induce slight deformation on the samples; furthermore, every time the thermocouples are repositioned, the distance between them lightly changes. The other parameters were easier calculated: ΔT was provided by thermocouples, while the thermal power was estimated as

$$P = V * I \tag{8}$$

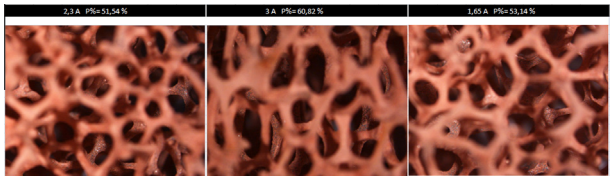

Fig. 2. The image analysis in three different sets of experiments: I = 1.65 A, 2.3 A and 3 A; P% = 50–60%.

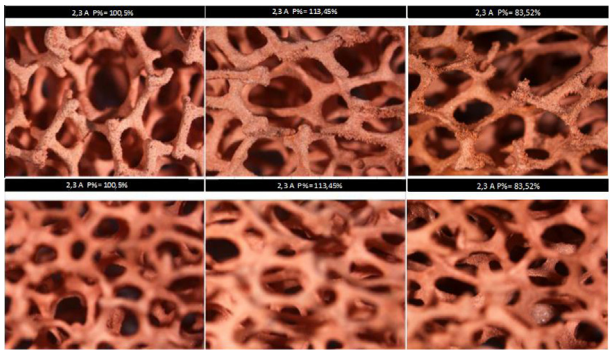

Fig. 3. The image analysis of samples with $P\%$ = 83.52%, 100.49% and 113.45%; I = 2.3 A.

4. Results

Here are showed the image analysis results acquired in three different sets of experiments, each one characterized by the different imposed values of $P\%$ and current. The test duration is instead found as a consequence of the choice of the previous parameters, exploiting the following relation:

$$t = (1/3.297 * 10^{-7}) * P * (\rho/\mathrm{i}) * V_i \tag{9}$$

For the first set (Fig. 2), were imposed P% in the range 50–60% and three values of current: 1.65 A, 2.3 A and 3 A. As showed by the images, the covering is insufficient, with an important lack of copper in particular within the foams. However, it is noticed that the best result was achieved with 2.3 A, while the worst with 1.65 A even do an higher $P\%$ (60%). Better results were obtained increasing $P\%$ until around 100%. In the second set (Fig. 3), were tested samples with this standard of covering, and in particular 83.52%, 100.49% and 113.45%, with 2.3 A. Although the first one resulted still insufficient, the other two displayed the formation of a homogenous cupric layer that covers entirely the aluminum structure, without voids. Based on the different density of the specimen considered, were obtained test durations in the range $2.7 * 10^3$–$4.5 * 10^3$ s, that represents an important improvement in comparison with those observed in absence of superficial treatments. In fact, as described in [19], the durations of electrodepositions necessary to obtain homogeneous coatings are much longer, up to $1.47 * 10^4$ s.

Finally, in the last test set P% was imposed in the range 144–155% exploiting 1.65 A, 2.3 A and 3 A (Fig. 4). It is noticed that only the specimen obtained with 2.3 A is perfectly electrodeposited. The others are unsatisfactory: in both cases, in fact, the cupric layer shows voids in the inner zones of the foams although the elevated quantity of deposited copper. The reasons of these behaviors are equal to those pointed out in not-pretreated foams: the use of high values of direct current (DC) causes a preferential deposition of copper on the external sides of the foams, because of the strong electric forces generated by the sample and, moreover, a

Table 1
Mechanical characteristics of the tested samples.

Density (kg/m^3)	P%	Y Strength (MPa)	Y Strain	Densification Strength [MPa]	Densification Strain
184.60	0.00	0.733	0.054	0.627	0.635
249.45	51.54	0.778	0.072	0.981	0.675
377.23	144.55	1.049	0.089	1.491	0.818
378.40	153.92	1.236	0.097	1.311	0.737
279.03	83.52	1.009	0.045	1.009	0.807
302.63	106.56	0.770	0.087	1.321	0.713
265.8	100.48	0.544	0.067	0.900	0.735
327.7	106.72	0.859	0.067	1.235	0.798
279.95	70.98	0.968	0.029	1.005	0.791
323.1	102.30	1.166	0.079	1.084	0.791

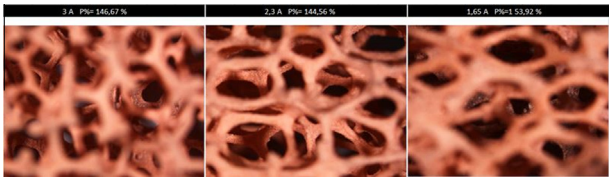

Fig. 4. The image analysis in three different sets of experiments: *I* = 1.65 A, 2.3 A and 3 A; *P*% = 144–155%.

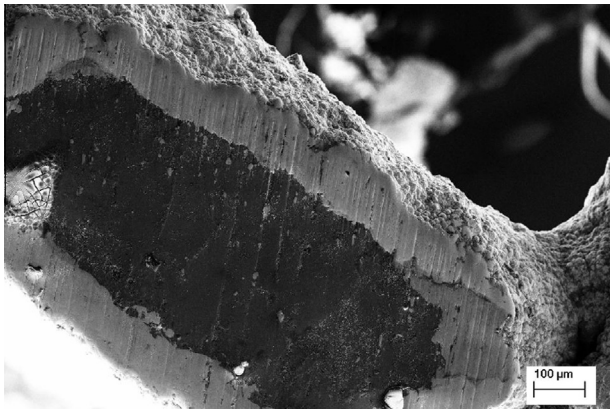

Fig. 5. SEM image.

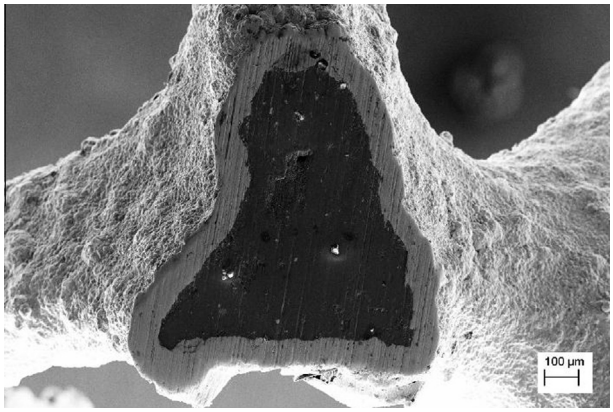

Fig. 6. SEM image.

progressive occlusion of the passages towards the inner part of the structure; low DC, instead, do not generate forces strong enough to guarantee a correct deposition.

In conclusion, it was established that, for samples of the considered shape and dimensions, a homogenous and satisfactory covering can be obtained with a value of *P*% that lays in the range 100–113%, and an average duration test of 1 h. Furthermore, considering the effects of I in the first and last test sets, better results can be achieved exploiting current value of 2.3 A.

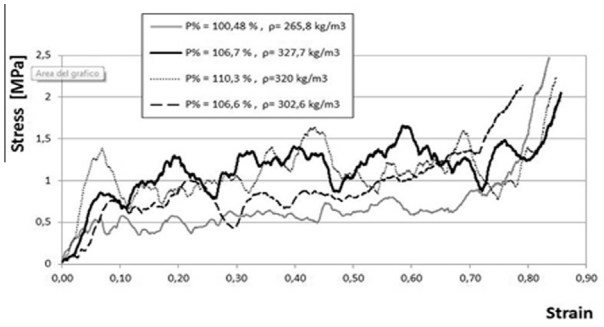

Fig. 7. The stress–strain curves.

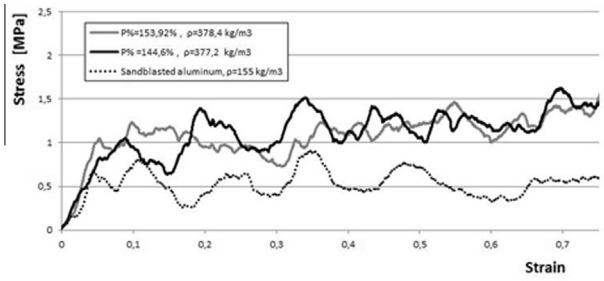

Fig. 8. The stress–strain curves.

SEM analysis leaded on *P*% = 100% samples, showed a perfect adhesion between the substrate and the covering layer as demonstrated in the following images (Fig. 5 and Fig. 6).

In the Fig. 7 are displayed some of the stress–strain curves obtained with compression test. According to the literature, three areas can be detected easily: an initial elastic zone, a plateau zone and an ending zone where occurs the failure of the material. Furthermore, it can be observed a sliding up of the mentioned curves proportionally to the density of the respective samples, whereas *P*% has no influence on this test, as better demonstrated in the Fig. 8. In fact, this parameter is related to the mass of the samples, which of course varies from specimen to specimen.

It was also highlighted that the behaviors of specimens with values of density higher than 320 kg/m^3 are very similar among them, showing an upper limit of resistance.

All these results are clearly summarized in the Fig. 9, where is plotted the deformation energy absorbed by the samples in function of the density of the specimens.

Table 2
The main characteristics and parameters of the samples subject to thermal conductivity tests.

Sample	Density (kg/m^3)	Thickness (m)	Voltage (V)	Current (A)	*P*%	Power (w)	ΔT (K)	$\lambda * S$ (W * m/k)
1	164.18	0.03	8.0	0.69	–	5.48	40.38	0.00402
1C	382.62	0.03	8.0	0.71	100.00	5.68	25.0	0.00673
2	179.27	0.0311	8.0	1.06	–	8.48	48.077	0.0054855
2C	351.57	0.0313	8.0	1.18	103.88	9.44	32.692	0.009038
3	203.49	0.0304	8.0	1.11	–	8.88	48.077	0.005615
3C	381.5	0.0306	8.0	1.2	97.80	9.6	32.692	0.0089856
4	184.89	0.030	8.0	1.18	–	9.44	50.00	0.005664
4C	360.0	0.0307	8.0	1.12	105.24	8.96	29	0.00961
5	183.3	0.0301	8.0	1.06	–	8.48	41.923	0.0060885
5C	361.18	0.0303	8.0	1.06	107.52	8.48	24.615	0.0104384

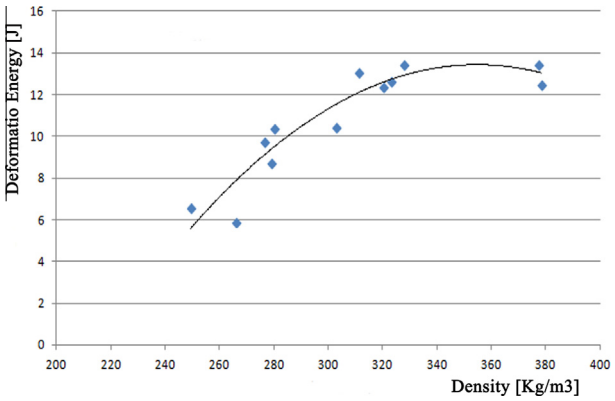

Fig. 9. The deformation energy absorbed in function of the density of the specimens.

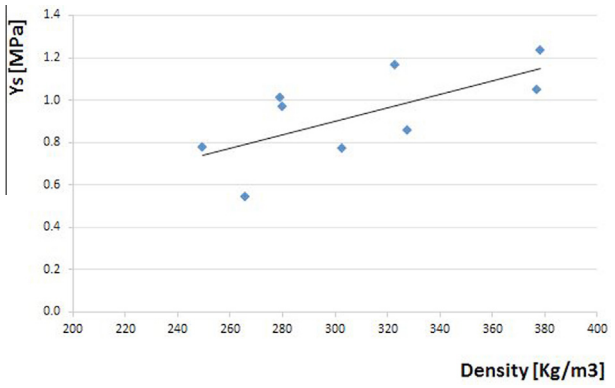

Fig. 10. The yield strength.

As it can be seen, deformation energy increases proportionally to the density of the samples, as a result of the enhancement of resistance of the material. Over 320 kg/m^3 the stress-strain curves are very similar, and it is observed a maximum of energy deformation of 14 J, at around 350 kg/m^3.

About the values of yield strength (Fig. 10), it is noticed a trend alike to the energy deformation one: it increases with the density of the samples. In this case, however, a greater dispersion of data is resulted because of the peculiar cellular structure that characterized each specimens, and the influence of P%, which makes impossible to define a precise yield strength trend in function of density. In the Table 1 are showed the mechanical characteristics of the tested samples. With P%= 0% is indicated the aluminium foam.

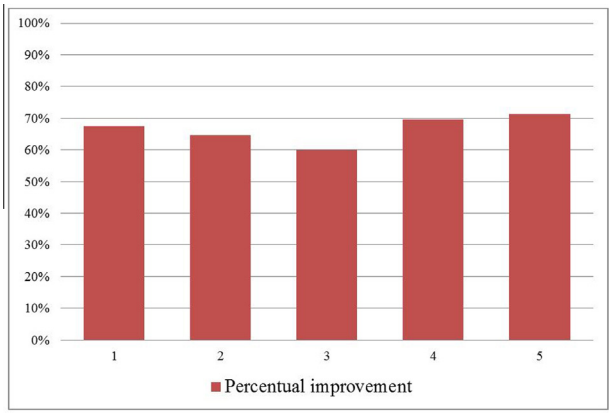

Fig. 11. The percentual improvement in thermal conductivity.

Supposing a linear trend, the calculator suggests a linear regression straight line of equation y = 0.0032x - 0.0504, and therefore the experimental results show a variance of 0.042 MPa.

In the Table 2 are showed the main characteristics and parameters of the samples subject to thermal conductivity tests. In particular, are mentioned thickness, density and *P*% of the different specimens, voltage and current exploited, the ΔT felt by the Peltier cells and, finally, the values assumed by the product $\lambda * S$, with which it is possible to calculate the percentual improvement in thermal conductivity, as demonstrated in the "Theoretical model" paragraph. With the letter "C" is indicated the sample after the electrodeposition process.

As it can be seen in the bar chart (Fig. 11), this enhancement is assessed in the range 60–70% for the considered specimens, which are characterized by a *P*% of around 100%.

5. Discussion

As already said, there are only a few works in literature that focus on the effects of coatings on mechanical and thermal features of aluminum foam substrates, and the procedure of electrodeposition itself. In [19] is completely described the experimental apparatus used in the experiments and the composition of the electrolytic bath, which are identical of those exploited in this work. Identical are also the value of exploited current and the model of electrdeposition (based on Faraday's law) used in both works, and the results match perfectly with what expected. A strong point of difference is in the surface preparation of the samples: in [19] in fact, this treatment is absent, and this leads to longer durations of electrodeposition. In this work has been demonstrated that the duration of the covering process, in order to obtain an homogeneous coating, passes from 1.47 $*$ 10^4 s

achieved in [19] to, depending on the densities of the specimens, $2.7 * 10^3$– $4.5 * 10^3$ s, which represents a very important improvement.

[17] enters more in what concerns compression tests. It is actually pointless to highlight the numerical differences found in test results because the experimental methods are very different. First of all , in [17] is used a pyrophosphate bath, working at pH 7.5 and kept at 65°C, and the samples were treated with a chemical process. In this work, an acid bath working at ambient temperature is used, and a sandblasting process was exploited as superficial treatment. Furthermore, and most important, the samples are shaped with different dimensions, and different speeds for compression test were used. Because of this, since the mechanical improvement is a strong function of foam topology, density and coating thickness, is more useful to put in evidence the similarities of general trends showed by the results. They show that the mechanical properties in terms of stiffness and plateau enhance with the thickness of the coating, as also the energy absorption capacity. Furthermore, it has been often verified that the collapse stress is higher in more covered samples than less ones, and also that, after elastic region, most of the samples showed a drop in resistance. All these trends are quite general and verified in other works like [7].

As the results demonstrates, the coating promote an enhancement of thermal conductivity that is assessed in the range 60–70%. In the past years, some investigations have been conducted for the thermal transport in metal foams for practical applications. [21,22,23]. Mancin et al [21] found a correlation between the heat transfer coefficient and the pressure drop. Bhattacharya et al [22] proposed an analytical and experimental investigation for the determination of the effective thermal conductivity, the permeability and inertial coefficient of high porosity metal foams. Fourie et al [23] presented a theoretical model for the prediction of pressure drop in a Newtonian fluid flowing through highly porous, isotropic metallic foams. Lu et al. [24] analyzed the forced convection problem in a tube filled with a porous medium subjected to constant wall heat flux. Finally a detailed study of forced convection in metal foams has been performed by Calmidi and Mahajan, [25]. All these studies assessed a potential improvement of the thermal exchange phenomena in systems fabricated with open cell foams components. In this work further improvements of the properties of heat transport in these systems were obtained.

6. Conclusions

In this work was studied the electrodeposition of copper on open cell aluminum foams, and the effect of a surface treatment of sandblasting on the process itself. Exploiting a model of deposition previously validated, it was established that, for samples of the considered shape and dimensions, a homogenous and satisfactory covering can be obtained with a value of *P%* that lays in the range 100–113%, and an average duration test of $3.6 * 10^3$ s, exploiting current value of 2.3 A. Higher or lower values of DC lead to worse results, with a lack of continuity of the covering, as demonstrated by the image analysis.

Compression tests showed that the resistance and the energy deformation of the specimens is proportional to their density, while there is no relation with the *P%* parameter. However, over 320 kg/m^3 the enhancement decreases and all the stress–strain curves tend to uniform themselves.

Finally, it is assessed an enhancement of the thermal conductivity of the considered specimens in the range 60–70%, for samples characterized by a *P%* of around 100%.

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