Correlation between factors controlling preparation of porous copper via sintering technique using experimental design

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There are many factors which control the porosity of the final object during preparation of porous copper compact using PM (powder metallurgy) technique. The amount of filler material, its type, sintering temperature, sintering time and the pressure of compaction were recognized as the most important parameters. In this investigation, naphthalene was used as filler material during porous copper preparation. Also, the correlation between these parameters and the porosity degree of the final object was developed with the aid of 2^N factorial design experiments. It was found that the amount of naphthalene represents the most important parameter controlling the porosity degree in the final object.

Key words: Porous copper, Powder metallurgy, Porosity, Naphthalene, Factorial design

INTRODUCTION

Over the past few decades, metals containing a number of voids (pores) have been studied and interest is growing in development for different industrial applications. Since, the importance of developing of such materials arises from its unique properties of low density and high specific surface area [1-5]. These porous metals provide specialized products for applications, such as filtration, fluid flow control, self-lubricating bearing, battery electrodes, etc.

There are various processes that could be used in preparation of porous metal such as vapor deposition, solid- gas eutectic solidification (GASAR) method [6,7], casting, and powder metallurgy (PM) [8]. The PM process was proved to be the most economic, feasible and promising technique in manufacturing of porous metal parts. Also, with using such technique there are almost no constrain considering complexity of the outer shape and geometry of the final porous metal object. PM techniques comprise foaming of the precursor prepared by compacting of powdered metal or alloy; whereas the pore forming gas is developed during melting of this precursor from admixed foaming agent [9]. The powder must be thoroughly compacted in order to seal the particles of foaming agent. This avoids the premature release of the gas on heating. PM porous metal can be prepared with gradiently variable pore size and also with preferred orientation of pores. Unfortunately, the mechanical properties of such materials are in many cases very poor, particularly under tensile loading [10-12]. Accordingly, it is important to control the various parameters affecting the porosity, pore size and morphology as well as the mechanical properties of the produced object. These affecting factors are the sintering temperature, the sintering time, the pressure of compaction as well as the amount, type, size of the foaming agent.

The preparation of porous metal was an interesting subject for many investigators. Leong and Liu [13] used copper powder of 63µm average size mixed with Emultex D64, which is a water-based binder. This produced like a paste feedstock which was then compacted in a mold container to obtain rectangular specimens and sintered at 800°C and 1000°C. The authors indicated that the advantage of these sintered wicks appears to be attributed to the existence of smaller pores with the controllability of porosity, and pore size that optimize heat pipe performance.

Zhao et al. [14] illustrated that Cu foams with porosity in the range 50–85% and cell sizes in the range 53–1500µm, had been manufactured by blending, compaction and sintering of Cu and K_2CO_3 powders followed by removal of K_2CO_3.

Zhao et al. [15] found that the cell morphology and size of the final Al-foam closely match those of the NaCl particles which used as foaming agent particles. The green porosity decreased with increasing compaction pressure and NaCl/Al ratio, where the foam porosity is 2–4% higher than the initial volume percentage of NaCl.

Takage and Yamauchi [16] indicated that the sublimable solid substance powder could be added to form pores having a predetermined diameter of 1 to 600µm in the porous body. The kind of the sublimable substance is not particularly restricted, so far as it is easily sublimated at a temperature of 200 to 800°C without any substantial residue being left. At least one member selected from camphor, menthol and naphthalene is ordinarily used as the sublimable substance.

Laptev et al. [17] used ammonium bicarbonate and carbamide powders as the filler materials for producing porous Ti parts. As both carbamide and ammonium bicarbonate decompose at 200°C, the porous metal structure is prone to collapse before strong bonding between the metal particles is formed at the sintering temperature. It is also hard to control the cell shape because of the significant shrinkage involved. Another problem of this method is that the decomposition of the filler material release environmentally damaging gases.

This paper presents the results of a simple experimental designed (2^N factorial design experiment) used in studying the

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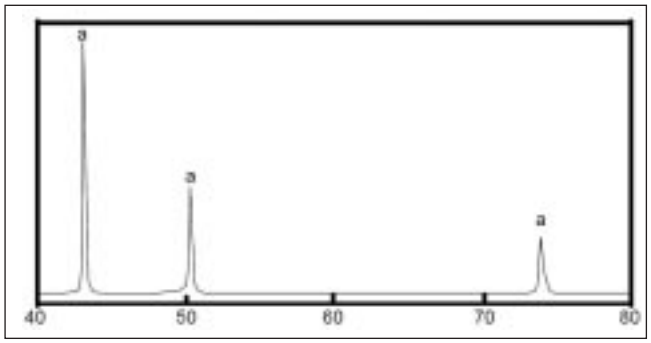

Fig. 1 – XRD of delivered copper powder (a = copper).

Fig. 1 – XRD della polvere di rame (a = copper).

effects of different controlling parameters on the preparation of porous copper metal compacts. Also, the correlation between the studied parameters and the degree of porosity, and pore morphology resulting in the final sintered object are investigated.

EXPERIMENTAL WORK

Raw Materials

Copper powder.

The copper powder used in this investigation was delivered from Ghatwary medical Borg El-Arab EL-Gedida (Egypt) Co. The grain size distribution of copper powder is shown in table 1 and the XRD Pattern of the copper powder was determined used Brucker AXS-D8 Advance and shown in Fig. 1, from which it is clear that the copper is the main element of this powder.

Naphthalene.

The naphthalene powder was delivered from O.S.S. laboratory of pure chemical powder of 128.17g/ml molecular weight. The particle size of naphthalene powder was found to be -160 + 125μm.

The Procedure

Copper powder was thoroughly mixed with varied amounts of Naphthalene powder. A certain amount of the produced mixture was uni-axially pressed with 75-300 MPa compaction pressure range to reach the desired height and diameter of 18 mm and 15 mm respectively for the compacted mixture. After compaction, the green copper compact is then heated for three hours at a temperature of 350°C to completely remove the naphthalene binder at this temperature (i.e. debinding). Then the temperature was allowed to rise to the desired sintering temperature at a rate of 5°C/min.

Equation (1) was used for evaluating the degree of porosity of the copper powder compacts [5].

$$\text{Porosity, \%} = \left(1 - \frac{\text{Apparent density of porous copper}}{\text{Density of non-porous copper}}\right) * 100 \qquad (1)$$

The apparent density of the individual specimen was calculated from a measurement of its weight and volume for each configuration.

Size μm	Percentage %
-200 +160	19.23
-160 +125	18.26
-125 +63	30.87
-63 + pan	31.74

Table (1) – Size distribution of copper powder.

Tabella (1) – Distribuzione delle dimensioni delle polveri di rame.

No. of Experiments	X1	X2	X3	X4	Y (results)
1	-	-	-	-	Y1
2	-	-	+	-	Y2
3	-	+	-	+	Y3
4	-	+	+	+	Y4
5	+	-	-	-	Y5
6	+	-	+	-	Y6
7	+	+	-	+	Y7
8	+	+	+	+	Y8

Table (3) – Matrix of eight experiments.

Tabella (3) – Matrice di otto esperimenti.

Experimental Design

In the present work the experimental design method (2^N factorial design) used to carry out a relationship between the different parameters affecting the porosity degree of the porous copper compacts. The factors controlling the porosity degree are sintering temperature, sintering time, compaction pressure and amount of naphthalene. Since the number of factors studied are four and according to the equation (2), for the 2^N factorial design, the number of experiments will be 16 experiments. Table (2) shows the level of the parameters, where X1 represent the sintering temperature, X2 represent the sintering time, X3 represent the compaction pressure and X4 is the amount of naphthalene. For the 16 experiments a matrix illustrated in Table (3). Table (4) will be used to describe the conditions of each experiment where Y will be the porosity degree obtained for each experimental condition. Equation (3) shows the general empirical formula describe the effect of a certain parameters (sintering temperature, sintering time, compaction pressure and amount of naphthalene) on a certain phenomena (the porosity degree of the sintered porous copper compacts). Where, the $\overline{X1}$, $\overline{X2}$, $\overline{X3}$ and $\overline{X4}$ are factors effecting on the degree of porosity of the sintered compacts and a_o, a_1, a_2, …… represents the coefficients of the equation it can be estimated from the Table (3) after carried out the experiments by multiplied the matrix of the results (Y) by each matrix of the factors. $\overline{X1}$, $\overline{X2}$ …, is called the X codes while the actual X could be obtained using equation (4).

No. of experiments $=2^N$,

where N= the no. of factors (2)

$$Y = a_o + a_1\overline{X1} + a_2\overline{X2} + a_3\overline{X3} + a_{12}\overline{X1}\,\overline{X2} + a_{23}\overline{X2}\,\overline{X3} + a_{13}\overline{X1}\,\overline{X3} + a_{123}\overline{X1}\,\overline{X2}\,\overline{X3} \qquad (3)$$

$$\overline{X_i} = \frac{X_i - X_o}{\Delta X_i} \qquad (4)$$

level	X1 (Sintering temperature, °C)	X2 (sintering time, min)	X3 (compaction pressure, MPa)	X4 (naphthalene amount, %)
Upper (+)	1000	60	300	7
Lower (-)	800	15	75	1
Average	900	37.5	187.5	4
interval	100	12.5	112.5	3

Table (2) – Levels of factors.

Tabella (2) – Livello dei fattori.

Where the Xi is the actual value of any factors, ΔXi is the interval showing in the Table (2) and X_o is the base value of X (factor) equal the average value of X.

RESULTS AND DISCUTION

Effect of naphthalene content and sintering temperature

It is worth to note that the proportion of copper powder and filler material was critical in order to produce a homogeneous and smooth feed stock [13]. Inadequate amount of the naphthalene in the mixture would cause a difficulty during molding, where the mixture would not flow easily and cracks would form during debinding due to the expansion of vapor trapped in the voids. So the adequate weight ratio of the copper powders to the naphthalene for producing sintered compacts having porosity from 20 to 50% should be determined experimentally.
Effect of the amount of naphthalene content on the total porosity of sintered copper compact produced at constant compaction pressure of 75 MPa and sintered at different temperatures (800-1000°C) while keeping the sintering time constant at 15 min is illustrated in Fig. 2. From these figure, it is clear that at any constant sintering temperature the increase of naphthalene content leads to an increase in the porosity of sintered copper compact because removal of naphthalene is accompanied by formation of pores. Thus the increase of naphthalene contents in the fresh mixture is responsible for the increase of the total porosity of the final sintered compacts.
The scanning electron microscope (SEM) micrographs shown in Fig. 3, of the sintered copper compacts produced at naphthalene content of 1% and 7% proved that increasing of the naphthalene contents leads to an increase in the total porosity of the final sintered compacts.

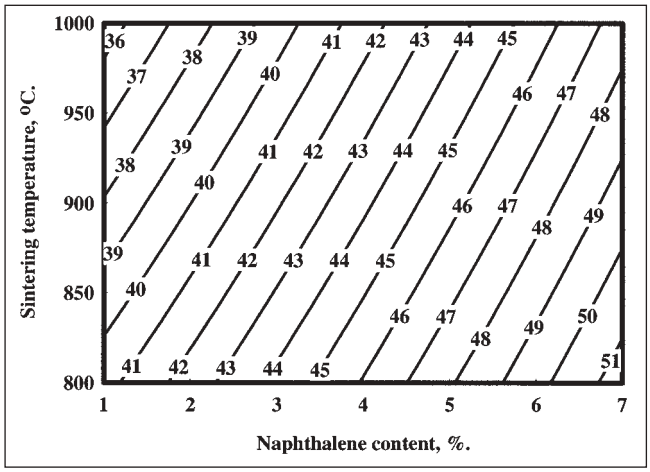

Fig. 2 – Effect of sintering temperature and naphthalene content on the total porosity of copper compact. (The compression load = 75 MPa, and the sintering time = 15 min.).

Fig. 2 – Effetto della temperatura di sinterizzazione e del contenuto di naftalene sulla porosità totale della polvere compattata. (Carico di compressione = 75 MPa. tempo di sinterizzazione = 15 min.).

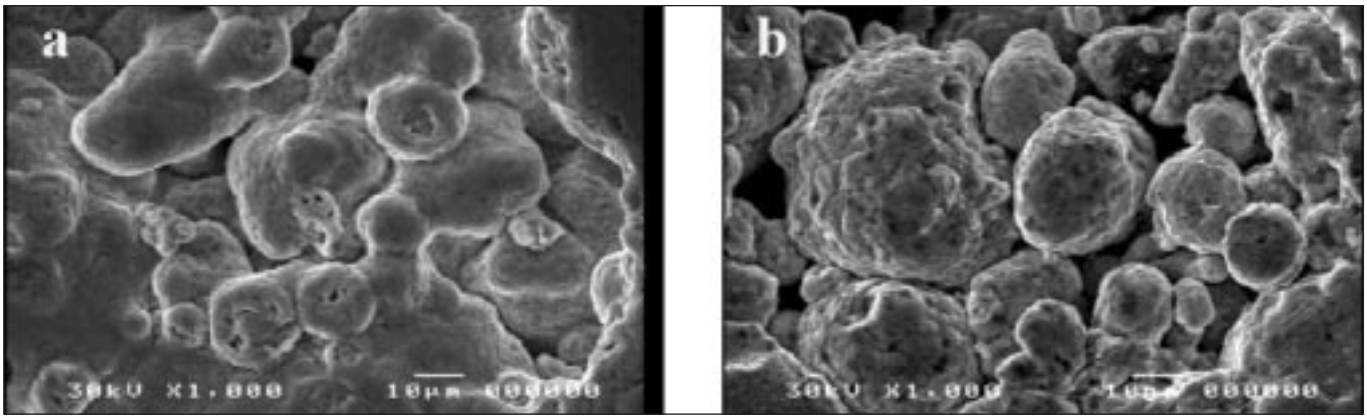

Fig. 3 – SEM micrographs for sinter copper compacts. (The sintering temperature 1000°C, sintering time 15 min. and compression load 75 MPa) a. 1% naphthalene; b. 7% naphthalene.

Fig. 3 – Micrografie SEM per i prodotti sinterizzati di rame. (Temperatura di sinterizzazione 1000°C, tempo di sinterizzazione 15 min., carico di compressione 75 MPa) a. 1% naftalene; b. 7% naftalene.

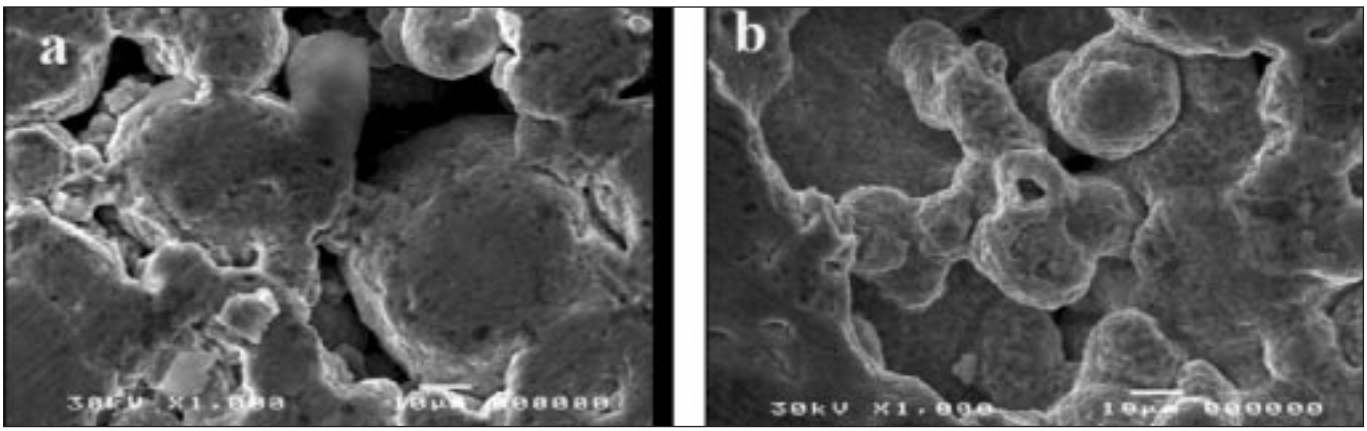

Fig. 4 – SEM micrographs for sinter copper compacts. (The amount of naphthalene added 7%, sintering time 15 min. and compression load 75 MPa) a. Sintered at 800°C; b. Sintered at 1000°C.

Fig. 4 – Micrografie SEM per i prodotti sinterizzati di rame. (Quantità di naftalene 7%, tempo di sinterizzazione 15 min., carico di compressione 75 MPa) a. Sinterizzato a 800°C; b. Sinterizzato a 1000°C.

On the other hand, selection of an appropriate sintering temperature is important during the processing of porous metal compact. It was found that when the sintering temperature was below 800°C, a completely intact compact could not be produced at even higher designated holding time of 60 min which leads to produce loose compacts. When the sintering temperature was above 1000°C some parts of the compact were softened which leads to inhomogeneous shape of the sintered compact. Accordingly, the sintering temperature used in this investigation was designed to be between 800-1000°C. Also, from Fig. 2 it is clear that at any constant amount of naphthalene content the increase of sintering temperature leads to a decrease in the total porosity of copper compacts. The decrease of the copper compact porosity with increasing sintering temperature could be attributed to the fact that, powder particles become extensively more collated [13]. Also it my attributed to that with increasing the sintering temperature a sound necks between powders is formed and their growth owing to the active mass transfer, giving rise to an increase in the contact areas and a decrease in the voids of the sample [18]. As shown in SEM micrograph, Fig. 4, gaps between particles are minimized and spaces left behind by the removal of naphthalene are filled up resulting in greater shrinkage.

The correlation between the total porosity of the sintered copper compacts (P %) and both of the sintering temperature (X_1, °C) and the naphthalene content (X4, %) when compaction pressure is 75 MPa and sintering time of 15 min is conducted and illustrated in the equation (5):

$$P = 60.27 - 2.68*10^{-2}X_1 + 1.03X_4 + 9.67*10^{-4}X_1X_4 \quad (5)$$

ffect of naphthalene content and compaction pressure

The selection of suitable compaction pressure during formation of porous metal compact is very important. During the formation of copper compacts it was noticed that, at compaction pressure below 75 MPa there was severe palling of copper particles yielding an imperfect geometry [15] and loose compact. Whereas at compaction pressure higher than 300 Mpa cracks were often induced in the samples, sometimes leading to complete fracture. Accordingly the range of compaction pressure between 75-300 MPa was applied in formation of copper compacts during this investigation.

The effect of both naphthalene content and compaction pressure on the total porosity of copper compact was carried out under constant sintering temperature of 800°C and constant sintering time of 15 minutes. The effect of both parameters on the total porosity is illustrated in Fig. 5. From these figures it is evident that at any constant amount of

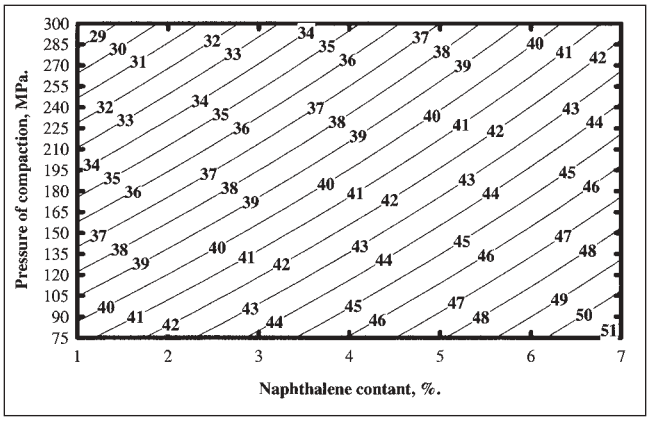

Fig. 5 – Effect of both naphthalene content and pressure of compaction on the total porosity of copper compacts. (The sintering temperature = 800°C and sintering time = 15 min.).

Fig. 5 – Effetto di contenuto di naftalene e pressione di compattazione sulla porosità totale della polvere compattata. (Temperatura di sinterizzazione = 800°C e tempo di sinterizzazione = 15 min.).

naphthalene the increase of compaction pressure leads to a decrease in the total porosity of the sintered copper compact. This may be due to the increase in the sintering rate with increased dislocated population. Also, it may be due to metal-metal contact formed during compaction, which was increased with increasing compaction pressure. These metal-metal contacts are fused together in the subsequent sintering process and interconnected in metallic framework and the expense of the voids present leads to a highly decrease in the total porosity. This phenomenon could be more clarified from the SEM micrograph as shown in Fig. 6. Also, at any constant compaction pressure the increased amount of naphthalene content makes the total porosity of the sintered copper compact to increase.

The correlation between the total porosity of sinter copper compact (P, %) and both the compaction pressure (X_3, MPa) and naphthalene content (X_4, %) is conducted and illustrated in the equation (6):

$$P = 43.22 - 5.82*10^{-2}X_3 + 1.66X_4 + 1.97*10^{-3}X_3X_4 \quad (6)$$

Effect of both naphthalene content and sintering time

The effect of sintering time and amount of naphthalene added on the total porosity of copper compacts were carried out at 800°C sintering temperature and 75 MPa compaction pressure.

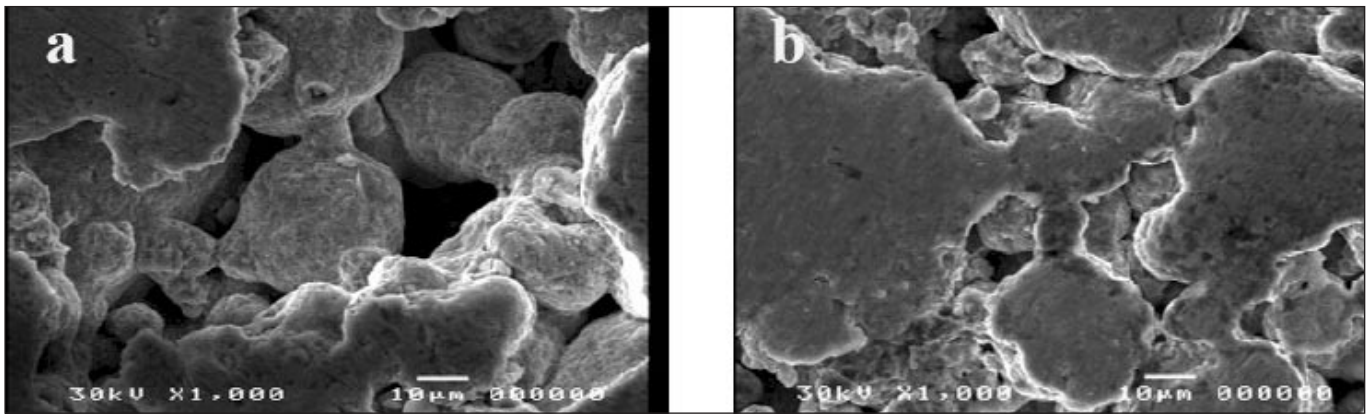

Fig. 6 – SEM micrographs for sinter copper compacts. (The sintering temperature 1000°C, sintering time 15 min. and Naphthalene content 7%) a. 75 MPa compaction pressure; b. 300 MPa compaction pressure.

Fig. 6 – Micrografie SEM dei prodotti di rame sinterizzato. (Temperatura di sinterizzazione 1000°C, tempo di sinterizzazione 15 min. e contenuto di naftalene 7%) a. pressione di compattazione 75 MPa; b. pressione di compattazione 300 MPa.

The effects of these parameters on the total porosity of copper compact are illustrated in Fig. 7. It is clear that at any constant amount of naphthalene content, the increase of sintering time is accompanied by a decreasing in the porosity of copper powder compacts [13]. This may be due to the fact that necking of copper particles is more pronounced with longer sintering times resulting in smaller pore size and consequently smaller total porosity. The SEM micrograph, Fig. 8, proves that, increasing the sintering time, the texture becomes more homogeneous and the grain growth increased at the expense of the pores formed as results of naphthalene evaporation. Also, at any constant sintering time the increase of amount of naphthalene added leads to a significant increase in the total porosity.
The correlation between both sintering time (X_2, min.) and naphthalene content (X_4, %) with the total porosity of copper compact (P, %) processed at constant temperature of 800°C with constant pressure of 75 Mpa, is expressed by the equation (7):

$$P = 39.27 - 2.79*10^{-2}X_2 + 1.84X_4 - 2.37*10^{-3}X_2X_4 \quad (7)$$

The general model for total porosity of porous copper compact

From the previous results, it was found that the general equation correlate the different controlling parameters with the porosity degree of porous copper compacts is expressed by equation (8):

$$P = 64.24 - 2.59*10^{-2}X_1 + 1.59*10^{-2}X_2 - 6.13*10^{-2}X_3 + 30.57*10^{-2}X_4 - 5.6*10^{-5}X_1X_2 + 8.8*10^{-6}X_1X_3 - 7.4*10^{-5}X_2X_3 + 1.52*10^{-3}X_1X_4 + 2.52*10^{-3}X_2X_4 + 1.91*10^{-3}X_3X_4 \quad (8)$$

Applications of this equation at the different experimental condition (as illustrated in table 3 as well as for extra four experiments out of the design (Table 5 shows the condition of the extra four experiments and the obtained porosity at each experiments)), for testing the validity of this equation in correlating the different sintering parameters as well as naphthalene content with the porosity degree of porous copper compacts was shown in Table 5. In this table the deviation between the experimental and theoretical values for the porosity degree was recorded. The deviation between the experimental and theoretical values was calculated using equation (9). From this table it could be concluded that the deviation between the experimental and theoretical results is ± 4% at most.

$$\text{Deviation, \%} = \left(\frac{\text{Theoretical value} - \text{Experimental value}}{\text{Theoretical value}}\right) * 100 \quad (9)$$

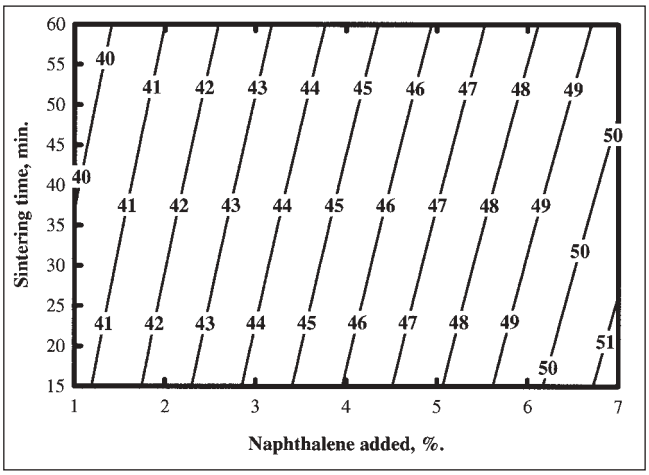

Fig. 7 – Effect of both sintering time and naphthalene amount added on the total porosity of copper compacts. (The sintering temperature = 800°C and the compaction pressure = 75 MPa).

Fig. 7 – Effetto di tempo di sinterizzazione e quantità di naftalene aggiunto sulla porosità totale della polvere compattata. (Temp. di sinterizzazione = 800°C e pressione di compattazione = 75 MPa).

Exp No.	X_1 Temp. (°C)	X_2 Time, (min.)	X_3 Compaction pressure, (MPa)	X_4 Naphthalene amount, (wt%)	Y Porosity degree, %
1	800	15	75	1	40.66
2	800	15	75	7	51.5
3	800	15	300	1	28
4	800	15	300	7	41.5
5	800	60	75	1	39.3
6	800	60	75	7	49.5
7	800	60	300	1	27
8	800	60	300	7	40
9	1000	15	75	1	35.5
10	1000	15	75	7	47.5
11	1000	15	300	1	25
12	1000	15	300	7	38.5
13	1000	60	75	1	34
14	1000	60	75	7	47
15	1000	60	300	1	20.63
16	1000	60	300	7	37

Table (4) – Description of each experiment condition and the degree of porosity obtained for each one.

Tabella (4) – Descrizione di ogni condizione sperimentale e grado di porosità ottenuta.

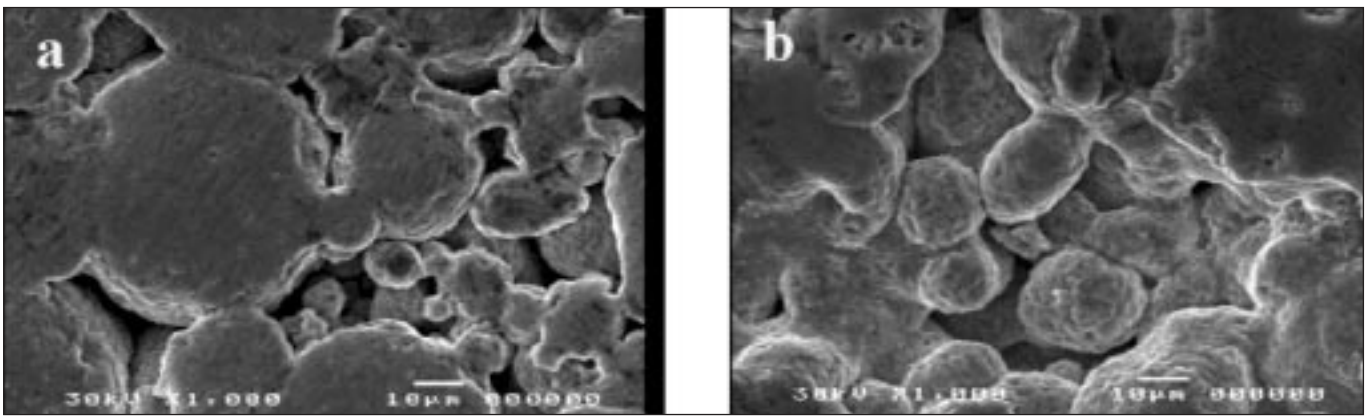

Fig. 8 – SEM micrographs for sinter copper compacts. (The sintering temperature 1000°C, Compaction load 75 MPa, and Naphthalene content 7%) a. sintering time, 15 min. b. sintering time, 60 min.

Fig. 8 - Micrografie SEM per i prodotti di rame sinterizzato. (Temperatura di sinterizzazione = 1000°C, pressione di compattazione = 75 MPa, e contenuto di Naftalene 7%) a. tempo di sinterizzazione, 15 min. b. tempo di sinterizzazione, 60 min.

Exp No.	X_1 Temp. (°C)	X_2 Time, (min.)	X_3 Compaction pressure, (MPa)	X_4 Naphthalene amount, (wt%)	Y Porosity degree, %
1	950	30	75	3	42
2	950	30	150	3	37
3	950	30	225	3	33
4	950	30	300	3	30.5

Table (5) – Description of extra four experiment condition out of the design and the degree of porosity obtained for each one.

Tabella (5) – Descrizione di quattro ulteriori condizioni sperimentali e grado di porosità ottenuta.

Exp No.	Experimental Porosity	Theoretical Porosity (P)	Deviation, %
1	40.66	40.65	-.025%
2	51.5	51	-0.97%
3	28	28.6	+2%
4	41.5	41.45	-0.1%
5	39.3	39.22	-0.2%
6	49.5	50.1	+1.2%
7	27	26.43	-2.1%
8	40	39.95	-0.1%
9	35.5	35.75	+0.7%
10	47.5	47.8	+0.6%
11	25	24.2	-3.2%
12	38.5	38.77	+0.7%
13	34	33.81	-0.55%
14	47	46.58	-0.89%
15	20.63	21.4	+3.7%
16	37	36.8	-0.5%
17	42	40.32	-4%
18	37	36.6	-1%
19	33	32.9	-0.3%
20	30.5	29.2	-4%

Table (6) – Differentiation between experimental results values and theoretical values.

Tabella (6) – Differenziazione fra valori sperimentali ottenuti e valori teorici.

CONCLUSION

From the obtained experimental results the following conclusions can be drawn:

1- Each of naphthalene content, sintering temperature, sintering time and compaction pressure, affect to a large extent, both the total porosity of the final sintered object and its morphology.
2- The amount of naphthalene; as filler material could be recognized as the most effective parameter during the preparation of porous copper compacts.
3- Using of an experimental design program (i.e. JUMPIN program) is an effective tool for investigating of the effects of these different parameters on the total porosity of the porous copper, and the deviation between the theoretical value and experimental results is in an acceptable range.

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ABSTRACT

RELAZIONE FRA FATTORI CHE CONTROLLANO LA PREPARAZIONE DI RAME POROSO MEDIANTE TECNICA DI SINTERIZZAZIONE CHE UTILIZZA DESIGN SPERIMENTALE

Parole chiave: Rame e sue leghe, metallurgia delle polveri, caratterizzazione materiali

Esitono diversi fattori che controllano la porosità finale del prodotto durante la preparazione del rame poroso mediante compattazione di polveri metalliche. La quantità e il tipo di filler, la temperatura, il tempo di sinterizzazione, la pressione di compattazione sono tutti parametri molto importanti. In questo studio è stato utilizzato il naftalene come filler per la preparazione di rame poroso. Inoltre è stata valutata la correlazione fra questi parametri e il grado di porosità del prodotto finale con l'aiuto di un programma sperimentale di design denominato JUMPIN. Si è osservato che la quantità di naftalene rappresenta il più importante parametro che controlla il grado di porosità del prodotto finale.