Ministry of Higher Education

And Scientific Research

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Sintering And production of A porous Material

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Fourth stage

Graduation project

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إهداء

بِسم الله الرَحمن الرحِيم

إلى كل من علموني واستَفدتُ منهم في حياتي العلمية والعملية في مقدمتهم اساتذتي ووالداي .

لهم جميعًا كل حُبي واعترافي بالجَميل .

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Introduction

Lightening a structure through the use of holes is a rather ancient concept that naturally led to the development of the new porous structures we are seeing today. A pore within a solid can be defined as a material containing pores (voids) . [1]

The shape and size of the voids have significant influence on the response of the material to external stimuli. The ability to describe the behavior of these materials has enabled engineers to tailor the macromechanical properties of porous materials to specific requirements. This has considerably promoted the applicability of such structures, which are playing an increasingly important role in several high impact sectors (aerospace, automotive, medical devices, etc.).This can be attributed to their unique combination of properties such as permeability, specific surface area, and biomimetic properties. [2]

porous material can also used as a self-lubricant material especially for some hidden areas which can not be lubricated manually , this process can be done by the immersion of the porous material in the lubricant so it remains trapped inside the pores , The flow of fluids through a porous media is a common topic of interest, and a separate studies has emerged called the study of general behavior of porous media. [3]

We can consider the existence of the pores as an advantage or disadvantage according to the application , for example porosity in casting considered as an unpleasant feature because of the corrosion effect as well as reduces the mechanical Properties such as strength , hardness , etc. But it is an important feature in some of the biomedical application because it enhances the

Osseointegration . Therefore, they have attracted the attention of both the industry and academics and have been the subject of intensive research in recent years. In terms of composition, porous materials can be inorganic, organic, or inorganic–organic composite materials (ceramics, glasses, metals, polymers, and their composites). In addition, recent advances in porous materials include the development of materials and shaping in the desired porous shapes. Moreover, tailoring of the total and type of porosity, pore size, and pore wall surface chemistry of porous materials are of scientific and technological importance . (4)

Porous materials are fundamental in a wide range of applications (e.g., catalysis, adsorption separation, sensing, drug delivery, biomedicine, energy production, energy storage systems, thermal and acoustic insulators).

there are several methods we can use to produce a porous material , in this research we will be using the powder metallurgy one .(5)

1.1 Powder Metallurgy PM

Powder metallurgy is a metal-forming process performed by heating compacted metal powders to just below their melting points. the process has become widely recognized as a superior way of producing high-quality parts for a variety of important applications . PM processes can reduce or eliminate the need for subtractive processes in manufacturing, lowering material losses and reducing the cost of the final product.(6)

Powder metallurgy is also used to make unique materials impossible to get from melting or forming in other ways. A very important product of this type is tungsten carbide (WC) . WC is used to cut and form other metals and is made from WC particles bonded with cobalt it is very widely used in industry for tools .(7)

The history of powder metallurgy and the art of metal and ceramic sintering are intimately related to each other. Sintering involves the production of a hard solid metal or ceramic piece from a starting powder. The ancient Incas made jewelry and other artifacts from precious metal powders. much wider range of products can be obtained from powder processes than from direct alloying of fused material.(8)

Powder metallurgy is an efficient and versatile advanced manufacturing process for both ferrous and non-ferrous parts. Powders are fed into a die and compacted under pressure. The resulting shapes are then sintered in a furnace to bond the particles together, a process that produces very little waste.

With the latest powder metallurgy manufacturing techniques, the scope of what is possible continues to grow and expand. Powder metallurgical engineering allows for the manufacturing of complex and unique shapes that simply aren’t possible with other metalworking processes.

1.2 Powder production techniques

Any fusible material can be atomized. Several techniques have been developed which permit large production rates of powdered particles, often with considerable control over the size ranges of the final grain population. Powders may be prepared by crushing, grinding, chemical reactions, or electrolytic deposition. The most commonly used powders are copper-base and iron-base materials.[9]

Here are two main powder metallurgy techniques :

1.2.1 Atomization

Atomization is accomplished by forcing a molten metal stream through an orifice at moderate pressures. A gas is introduced into the metal stream just before it leaves the nozzle, serving to create turbulence as the entrained gas expands (due to heating) and exits into a large collection volume exterior to the orifice. The collection volume is filled with gas to promote further turbulence of the molten metal jet. Air and powder streams are segregated using gravity or cyclonic separation.

Most atomized powders are annealed, which helps reduce the oxide and carbon content. The water atomized particles are smaller, cleaner, and nonporous and have a greater breadth of size, which allows better compacting. The particles produced through this method are normally of spherical or pear shape. Usually, they also carry a layer of oxide over them. [10]

There are three types of atomization:

- Liquid atomization
- Gas atomization
- Centrifugal atomization

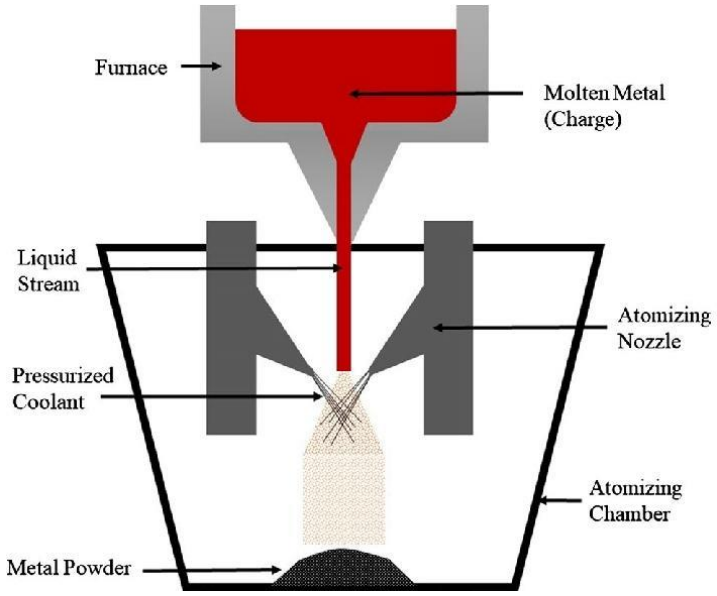


Figure (1-1) schematic for the Atomization process

1.2.2 Electrolytic method

It is used extensively in the preparation of copper, beryllium, iron and nickel powders. Adjustment of the chemical and physical conditions during electrodeposition makes it possible to cause the metal to deposit loosely on the cathode of the cell either as a light cake or in flake form. Both are easily crushed into a powder. The method yields a high purity metal with excellent properties for conventional powder metallurgy processing. The process involves the control and it may be very difficult to produce a high purity powdery deposit at relatively fast rates economically. Consequently, in many cases the deposit is a solid and must be pulverized, for example iron. Electrolytic deposits, powders or solids are usually very reactive and brittle. For both these reasons the material may be given a special annealing treatment. Powders formed during electrolysis have a characteristic dendritic shape[(11)]

1.3 Powder compaction

Powder compaction is the process of compacting metal powder in a die through the application of high pressures. Typically the tools are held in the vertical orientation with the punch tool forming the bottom of the cavity. The powder is then compacted into a shape and then ejected from the die cavity . In a number of these applications the parts may require very little additional work for their intended use; making for very cost efficient manufacturing.

The density of the compacted powder increases with the amount of pressure applied. Typical pressures range from 80 psi to 1000 psi (0.5 MPa to 7 MPa), pressures from 1000 psi to 1,000,000 psi have been obtained. Pressure of 10 t/in² to 50 t/in² (150 MPa to 700 MPa) are commonly used for metal powder compaction. To attain the same compression ratio across a component with more than one level or height, it is necessary to work with multiple lower punches. A cylindrical workpiece is made by single-level tooling. A more complex shape can be made by the common multiple-level tooling.(12)

1.3.1 Die pressing

The dominant technology for the forming of products from powder materials, in terms of both tonnage quantities and numbers of parts produced, is die pressing. There are mechanical, servo-electrical and hydraulic presses available in the market, whereby the biggest powder throughput is processed by hydraulic presses.

1.3.2 Isostatic pressing

In some pressing operations, such as hot isostatic pressing (HIP) compact formation and sintering occur simultaneously. This

procedure, together with explosion-driven compressive techniques is used extensively in the production of high-temperature and high-strength parts such as turbine disks for jet engines. In most applications of powder metallurgy the compact is hot-pressed, heated to a temperature above which the materials cannot remain work-hardened. Hot pressing lowers the pressures required to reduce porosity and speeds welding and grain deformation processes. It also permits better dimensional control of the product, lessens sensitivity to physical characteristics of starting materials, and allows powder to be compressed to higher densities than with cold pressing, resulting in higher strength. Negative aspects of hot pressing include shorter die life, slower throughput because of powder heating, and the frequent necessity for protective atmospheres during forming and cooling stages.[13]

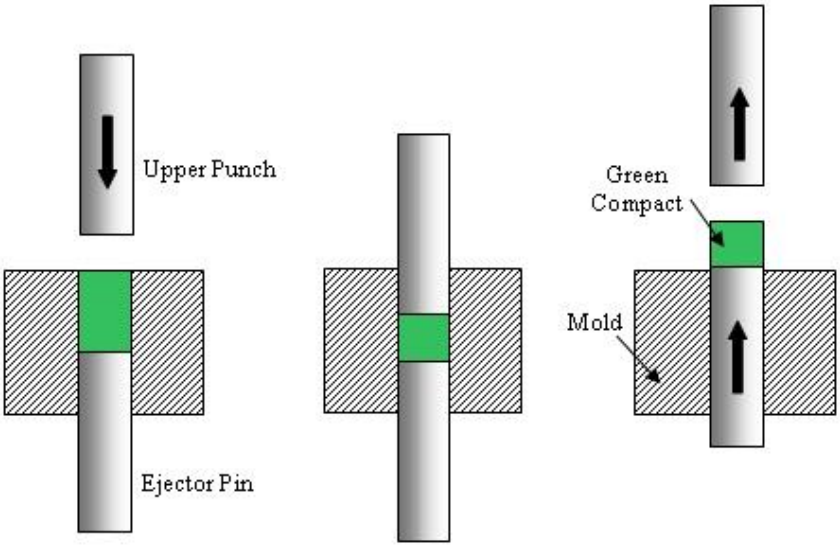


Figure (2-1) Compaction process

1.4 Sintering

Sintering of powder metals is a process in which particles under pressure chemically bond to themselves in order to form a coherent shape when exposed to a high temperature. The temperature in which the particles are sintered is most commonly below the melting point of the main component in the powder. If the temperature is above the melting point of a component in the powder metal part, the liquid of the melted particles fills the pores. This type of sintering is known as liquid-state sintering. (14)

A major challenge with sintering in general is knowing the effect of the process on the dimensions of the compact particles. This is especially difficult for tooling purposes in which specific dimensions may be needed. It is most common for the sintered part to shrink and become denser, but it can also expand or experience no net change.

The main driving force for solid state sintering is an excess of surface free energy. The process of solid-state sintering is complex and dependent on the material and furnace (temperature and gas) conditions. (15)

There are six main stages that sintering processes can be grouped in which may overlap with one another :

1. initial bonding among particles .

2. neck growth .

3. pore channel closure .

4. pore rounding .

5. densification or pore shrinkage .

6. pore coarsening.

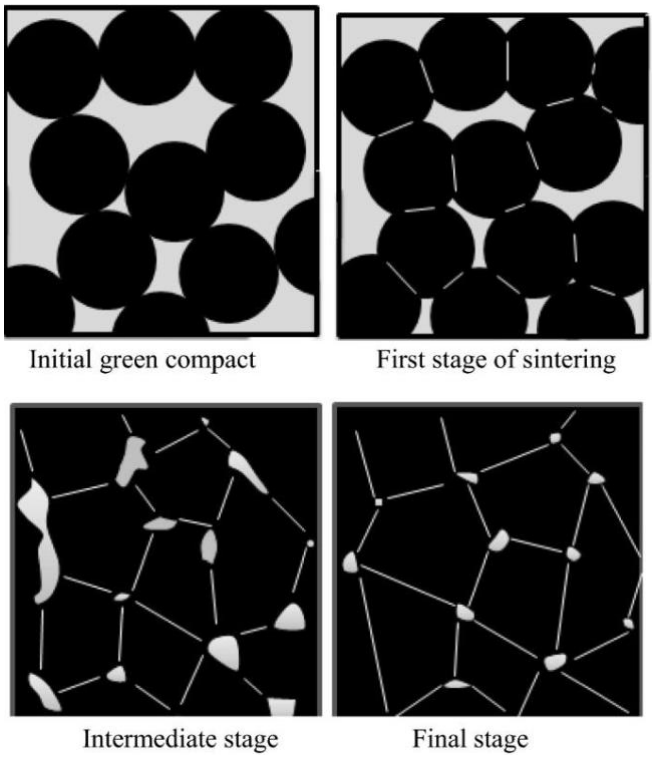


Figure (3-1) sintering stages

Most sintering furnaces contain three zones with three different properties that help to carry out the six steps above.

The first zone, commonly coined the burn-off or purge stage, is designed to combust air, burn any contaminants such as lubricant or binders, and slowly raise the temperature of the compact material .

The second zone, known as the high-temperature stage, is used to produce solid-state diffusion and particle bonding. The material is seeking to lower its surface energy and does so by moving toward the points of contact between particles. The contact points become larger and eventually a solid mass with small pores is created.

The third zone, also called the cooling period, is used to cool down the parts while still in a controlled atmosphere. This is an

important zone as it prevents oxidation from immediate contact with the air or a phenomenon known as rapid cooling. (16)

During this process, a number of characteristics are increased including the strength, ductility, toughness, and electrical and thermal conductivity of the material. If different elemental powders are compact and sintered, the material would form into alloys and intermetallic phases.
As the pore sizes decrease, the density of the material will increase. As stated above, this shrinkage is a huge problem in making parts or tooling in which particular dimensions are required. The shrinkage of test materials is monitored and used to manipulate the furnace conditions or to oversize the compact materials in order to achieve the desired dimensions. Although, sintering does not deplete the compact part of porosity. In general, powder metal parts contain five to twenty-five percent porosity after sintering. (17)

1.5 Hazards of powder metallurgy

The special materials and processes used in powder metallurgy can pose hazards to life and property. The high surface-area-to-volume ratio of the powders can increase their chemical reactivity in biological exposures (for example, inhalation or ingestion), and increases the risk of dust explosions. Materials considered relatively benign in bulk can pose special toxicological risks when in a finely divided form. Inhalation of heavy metals can result in many health issues. Lead and cadmium are generally toxic, and cobalt can cause asthma and fibrosis in sensitive individuals.(18)

Part Two : Experimental work

Introduction

In this study which is used to produce a porous material by using the powder metallurgy techniques , two metals are selected , one with a low melting point (Zinc 419.5 C°) and the other with a relatively high melting point (Copper 1085 C°) they are being prepared by the popular steps of powder preparation to get four pellets with different percentages of copper and zinc .

Figure (1-2) Copper metal : Powder , pressing pellet with a small percentage of zinc

As it known , sintering is the most important step in powder metallurgy , when the samples are sintered in a high temperature which is for sure higher than the melting point of zinc , the zinc inside these samples will burn or evaporate leaving voids or pores behind , this way we can also control the size and number of the pores .

The greater the percentage of zinc in the sample is , the more voids or pores that are formed .

This will be proven by the following steps :

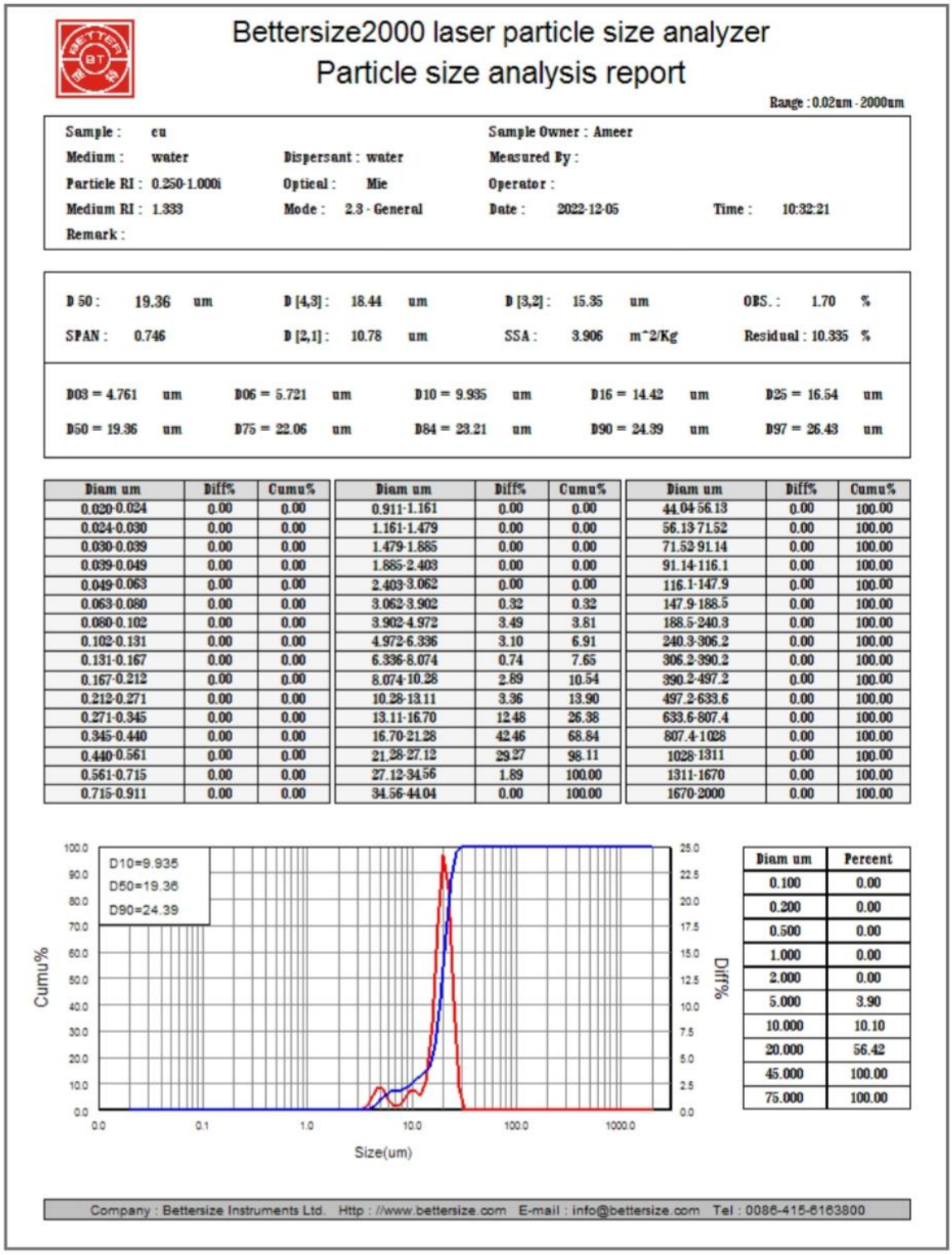
2.1 Preparation of powders

Table 1-2 : Particle size , Purity and Originality of the powders used (Cu , Zn)

Powders	Particle size	Purity	Originality
Copper	19.36 um	99.9 %	China
Zinc	25.67 um	99.9 %	China

Particle size for the material used (Copper and Zinc) is measured by Better size 2000 to make sure that they are different in size .

Figure (2-2) Better size 2000



Bettersize2000 laser particle size analyzer

Particle size analysis report

Range : 0.02um - 2000um

Sample :	cu			Sample Owner : Ameer	
Medium :	water	Dispersant :	water	Measured By :	
Particle RI :	0.250-1.000i	Optical :	Mie	Operator :	
Medium RI :	1.333	Mode :	2.3 - General	Date : 2022-12-05	Time : 10:32:21
Remark :					

D 50 :	19.36	um	D [4,3] :	18.44	um	D [3,2] :	15.35	um	OBS. : 1.70 %
SPAN :	0.746		D [2,1] :	10.78	um	SSA :	3.906	m^2/Kg	Residual : 10.335 %

D03 = 4.761	um	D06 = 5.721	um	D10 = 9.935	um	D16 = 14.42	um	D25 = 16.54	um
D50 = 19.36	um	D75 = 22.06	um	D84 = 23.21	um	D90 = 24.39	um	D97 = 26.43	um

Diam um	Diff%	Cumu%	Diam um	Diff%	Cumu%	Diam um	Diff%	Cumu%
0.020-0.024	0.00	0.00	0.911-1.161	0.00	0.00	44.04-56.13	0.00	100.00
0.024-0.030	0.00	0.00	1.161-1.479	0.00	0.00	56.13-71.52	0.00	100.00
0.030-0.039	0.00	0.00	1.479-1.885	0.00	0.00	71.52-91.14	0.00	100.00
0.039-0.049	0.00	0.00	1.885-2.403	0.00	0.00	91.14-116.1	0.00	100.00
0.049-0.063	0.00	0.00	2.403-3.062	0.00	0.00	116.1-147.9	0.00	100.00
0.063-0.080	0.00	0.00	3.062-3.902	0.32	0.32	147.9-188.5	0.00	100.00
0.080-0.102	0.00	0.00	3.902-4.972	3.49	3.81	188.5-240.3	0.00	100.00
0.102-0.131	0.00	0.00	4.972-6.336	3.10	6.91	240.3-306.2	0.00	100.00
0.131-0.167	0.00	0.00	6.336-8.074	0.74	7.65	306.2-390.2	0.00	100.00
0.167-0.212	0.00	0.00	8.074-10.28	2.89	10.54	390.2-497.2	0.00	100.00
0.212-0.271	0.00	0.00	10.28-13.11	3.36	13.90	497.2-633.6	0.00	100.00
0.271-0.345	0.00	0.00	13.11-16.70	12.48	26.38	633.6-807.4	0.00	100.00
0.345-0.440	0.00	0.00	16.70-21.28	42.46	68.84	807.4-1028	0.00	100.00
0.440-0.561	0.00	0.00	21.28-27.12	29.27	98.11	1028-1311	0.00	100.00
0.561-0.715	0.00	0.00	27.12-34.56	1.89	100.00	1311-1670	0.00	100.00
0.715-0.911	0.00	0.00	34.56-44.04	0.00	100.00	1670-2000	0.00	100.00

Diam um	Percent
0.100	0.00
0.200	0.00
0.500	0.00
1.000	0.00
2.000	0.00
5.000	3.90
10.000	10.10
20.000	56.42
45.000	100.00
75.000	100.00

Company : Bettersize Instruments Ltd. Http : //www.bettersize.com E-mail : info@bettersize.com Tel : 0086-415-6163800

Figure (3-2) Particle size analysis for Copper

Bettersize2000 laser particle size analyzer
Particle size analysis report

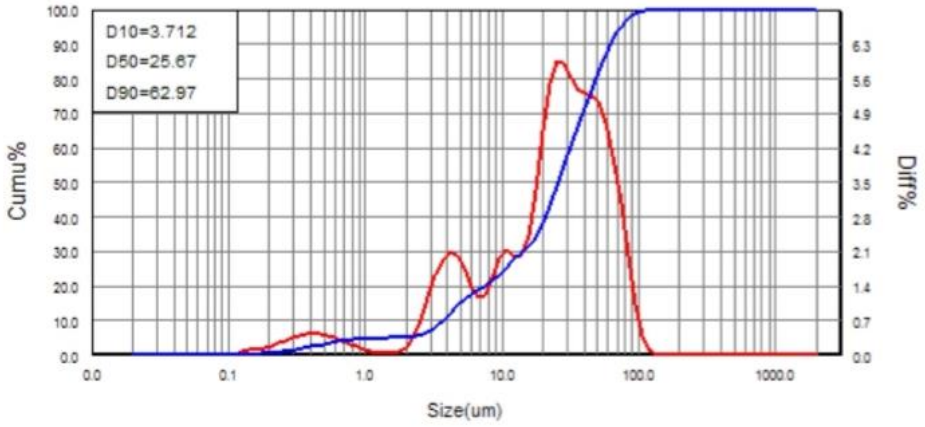
Range : 0.02um - 2000um

Sample :	Zn		Sample Owner : Bashaar		
Medium :	water	Dispersant : water	Measured By :		
Particle RI :	1.520-0.100i	Optical : Mie	Operator :		
Medium RI :	1.333	Mode : 2.3 - General	Date : 2022-11-17	Time :	06:18:43
Remark :					

D 50 :	25.67	um	D [4,3] :	29.86	um	D [3,2] :	4.903	um	OBS. : 8.39 %
SPAN :	2.307		D [2,1] :	0.411	um	SSA :	122.3	m^2/Kg	Residual : 3.421 %

D03 = 0.485 um	D06 = 2.645 um	D10 = 3.712 um	D16 = 5.331 um	D25 = 10.77 um
D50 = 25.67 um	D75 = 43.88 um	D84 = 53.98 um	D90 = 62.97 um	D97 = 81.11 um

Diam um	Diff%	Cumu%	Diam um	Diff%	Cumu%	Diam um	Diff%	Cumu%
0.020-0.024	0.00	0.00	0.911-1.161	0.21	4.76	44.04-56.13	10.41	85.56
0.024-0.030	0.00	0.00	1.161-1.479	0.08	4.84	56.13-71.52	8.50	94.06
0.030-0.039	0.00	0.00	1.479-1.885	0.09	4.93	71.52-91.14	4.66	98.72
0.039-0.049	0.00	0.00	1.885-2.403	0.52	5.45	91.14-116.1	1.20	99.92
0.049-0.063	0.00	0.00	2.403-3.062	1.85	7.30	116.1-147.9	0.08	100.00
0.063-0.080	0.00	0.00	3.062-3.902	3.52	10.82	147.9-188.5	0.00	100.00
0.080-0.102	0.00	0.00	3.902-4.972	4.18	15.00	188.5-240.3	0.00	100.00
0.102-0.131	0.04	0.04	4.972-6.336	3.18	18.18	240.3-306.2	0.00	100.00
0.131-0.167	0.19	0.23	6.336-8.074	2.42	20.60	306.2-390.2	0.00	100.00
0.167-0.212	0.31	0.54	8.074-10.28	3.60	24.20	390.2-497.2	0.00	100.00
0.212-0.271	0.53	1.07	10.28-13.11	4.18	28.38	497.2-633.6	0.00	100.00
0.271-0.345	0.73	1.80	13.11-16.70	4.57	32.95	633.6-807.4	0.00	100.00
0.345-0.440	0.85	2.65	16.70-21.28	8.20	41.15	807.4-1028	0.00	100.00
0.440-0.561	0.82	3.47	21.28-27.12	11.60	52.75	1028-1311	0.00	100.00
0.561-0.715	0.66	4.13	27.12-34.56	11.61	64.36	1311-1670	0.00	100.00
0.715-0.911	0.42	4.55	34.56-44.04	10.79	75.15	1670-2000	0.00	100.00



Diam um	Percent
0.500	3.10
1.000	4.65
2.000	5.00
5.000	15.07
10.000	23.72
20.000	38.64
45.000	76.07
75.000	95.26
100.000	99.52
200.000	100.00

Company : Bettersize Instruments Ltd. Http : //www.bettersize.com E-mail : info@bettersize.com Tel : 0086-415-6163800

Figure (4-2) Particle size analysis for Zinc

2.2 Sample preparation and weighting

Prepare four samples in different percentages (wt%) with 5g for each one using a sensitive balance :

Table 2-2 : Content of Cu and Zn in each sample

Sample NO.	Cu%	Zn%
1	80	20
2	60	40
3	50	50
4	20	80

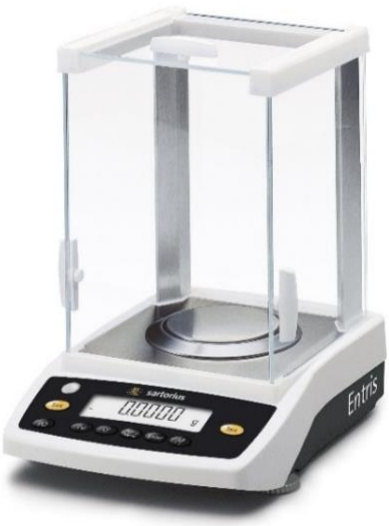


Figure (5-2) Sensitive balance

2.3 Compaction

After preparing the samples and doing the mixing process for 3 hours , we start with the pressing process using the Carver model 43874NE 0000 pellet press .

It is important to use lubrication to prevent sticking between the powder and the mold metal (Diameter 13mm – Thickness 5mm) Here ethanol was used as a lubricant .

Pressing the samples under 700Mpa (9.476 ton) and 4 min time for each sample .

Figure (6-2) Carver model 43874NE 0000 pellet press

2.4 Porosity and density test for the green samples

We can measure the porosity for green samples (before sintering process) by taking the Dry weight (D) , saturated weight (M) and hanging weight (S) for each sample then using the formula : Porosity = M-D/M-S

As for density we can measure it directly using Archimedes method .

Table 3-2 : porosity and density for the green samples

Sample NO.	Porosity %	Density g/cm^3
1	3.43	7.1624
2	3.66	7.0548
3	4.1	6.7711
4	8.15	6.676

2.5 Sintering

The samples are sintered by sintering furnace type GSL1600X using Argon gas because of its passivity to prevent the oxidation

Figure (7-2) Sintering furnace

Figure (8-2) Samples after the sintering process

Sintering procedure is Heating from the room temperature to 250 C° then soaking (holding) for one hour before heating again to 750 C° for two hour and half .

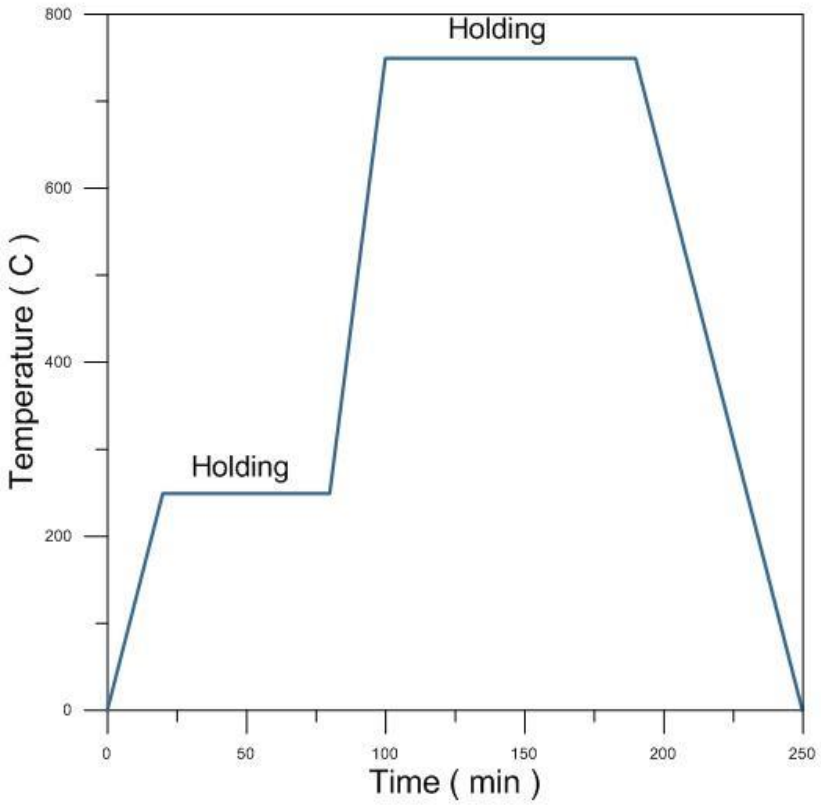


Figure (9-2) Sintering procedure

2.6 Porosity and density test for the sintered samples

As for the green samples (before sintering) we can measure the porosity and density for sintered samples by the same ways .

Table 4-2 : Porosity and density for sintered samples

Sample NO.	Porosity %	Density g/cm^3
1	21.18	6.338
2	31.07	5.567
3	39.6	4.773
4	40.7	3.608

2.7 Grinding and polishing

The samples are grinded using abrasive papers (180 , 220 , 400 , 800 , 1000 , 1500 , 2000) respectively .

Lubricant by fluids should be used during grinding - such as water - to reduce the friction rate .

Then start with the polishing process using a velvet polishing cloth and diamond paste for a smooth and shiny surface .

Figure (10-2) Samples after grinding and polishing process

Part three : Discussion

Introduction

In this part, we will discuss the results that we obtained from conducting the necessary tests, which include Particle size analysis , XRD analysis and porosity and density tests for each sample before and after the sintering process .

We also will be drawing the relationship between the percentage of zinc (Zinc%) with the porosity and density before and after sintering process for the purpose of showing the effect of volatilization or evaporation of zinc on each of them .

3.1 XRD Analysis

In order to make sure that the powder is copper, we conducted an X-ray test for the copper powder

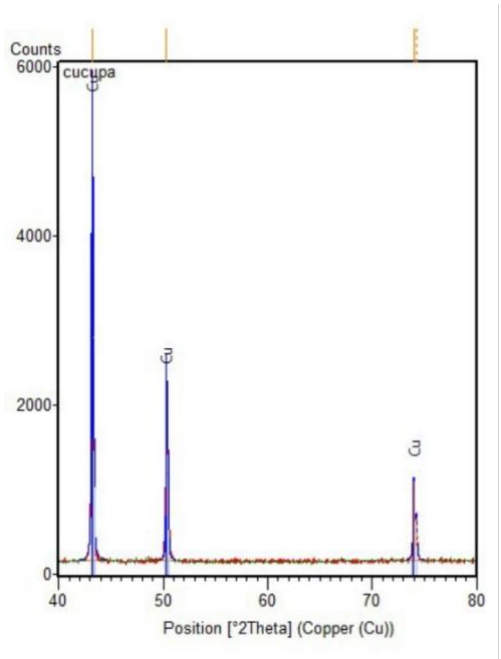


Figure (1-3) XRD analysis for Copper powder

After sintering, we conducted an X-ray test for sample 3 (50% Cu , 50% Zn) in order to identify the effect of the sintering process on the constituent metals of the sample (Cu , Zn) .

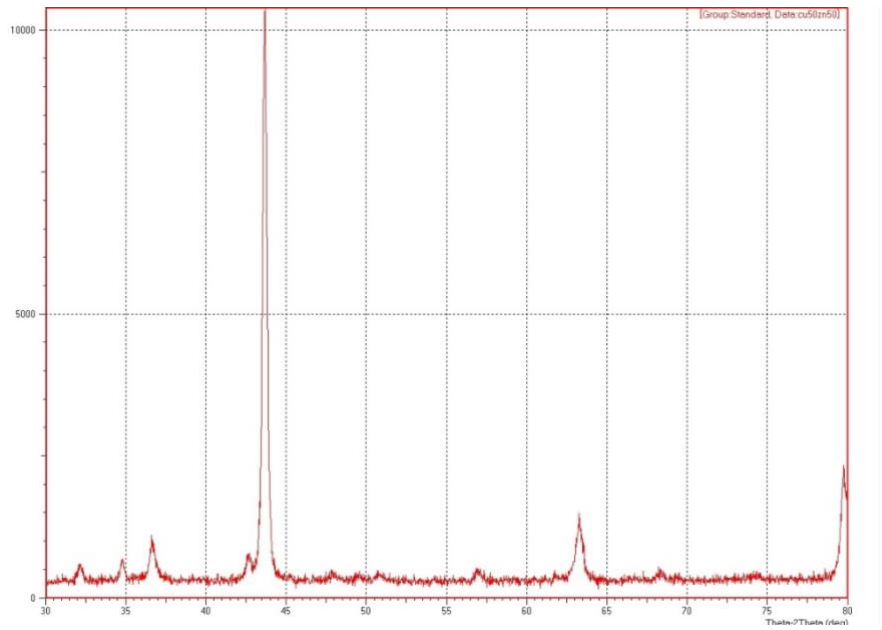


Figure (2-3) XRD analysis for (50% Cu , 50% Zn)

We notice from Figure (2-3) that the copper metal appeared in most values of 2 theta i.e. much more than the appearance of zinc , although the percentage of zinc in the sample is equal to the percentage of copper , this is due to the high evaporation of zinc during sintering .

3.2 Particle size

From Table (1-2), we notice that there is a good homogeneity in the sizes of the particles for both zinc and copper metals, as one of the principles of powder metallurgy is that we use different sizes (small, medium, large) to obtain homogeneity for the

sintering process to be successful because the small particles overlap between the large particles .

3.3 Porosity and density before and after the sintering

Before sintering , from Table (3-2) we note that there is a significant increase in porosity from sample No. 1 to sample No. 4 and this is expected for the green samples because the percentage of zinc in sample 4 (80%) is greater than that in sample 1(20%) , and this is due to the fact that the particle size of zinc (26um) which is the element with the largest percentage in the sample 4 is larger than the particle size of copper (19um).

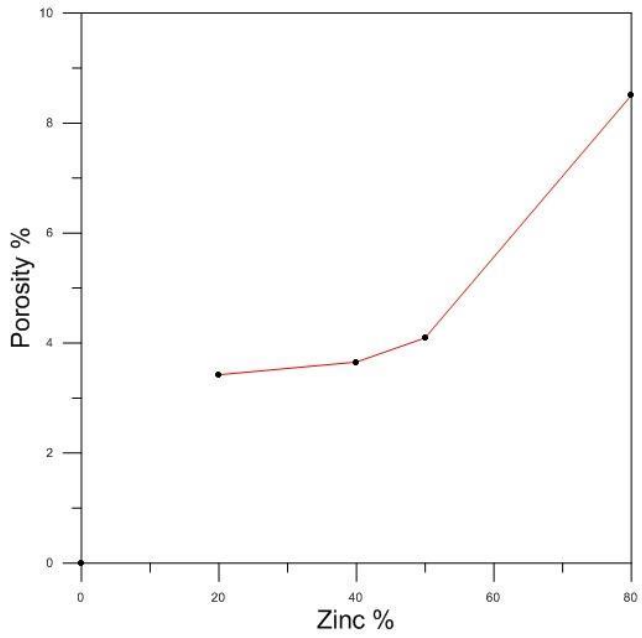


Figure (3-3) relationship between the Zinc % and Porosity % before sintering

Thus, the green density decreases in return, as there is an inverse relationship between density and porosity, as in the figure (4-3):

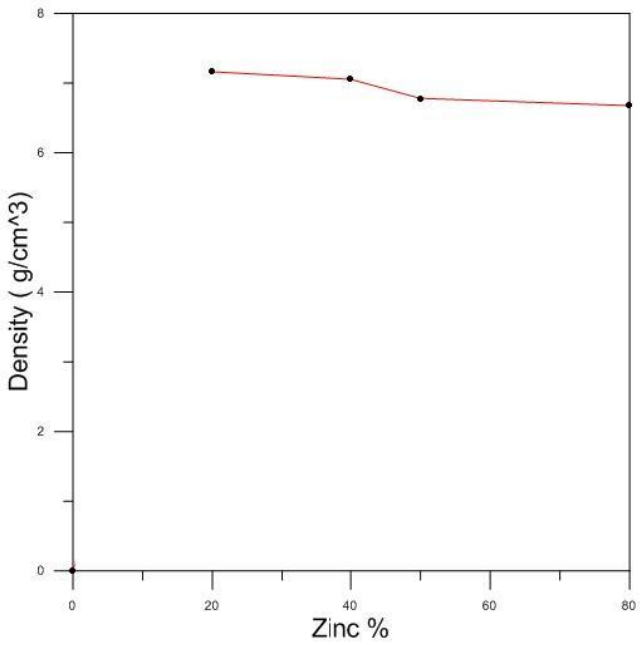


Figure (4-3) relationship between the Zinc% and density before sintering

After sintering , We note from Table (4-2) that after the sintering process and the occurrence of a bonding process between the atoms of the powders used, there is a significant increase in the porosity which is the opposite of what is known , this is due to the intense evaporation of zinc during the sintering process, as its evaporation left many voids .

The first stage of the sintering process is the main factor which can control the final value of the porosity of the samples. Accordingly, the duration of the first stage of sintering plays a main role in the process of the evaporation of the zinc element. As it is known, the boiling temperature of the zinc element at (760 mm Hg) is (907 degrees Celsius). This value is reduced dramatically under vacuum environment. This fact can be observed in Fig (5-3).[19]

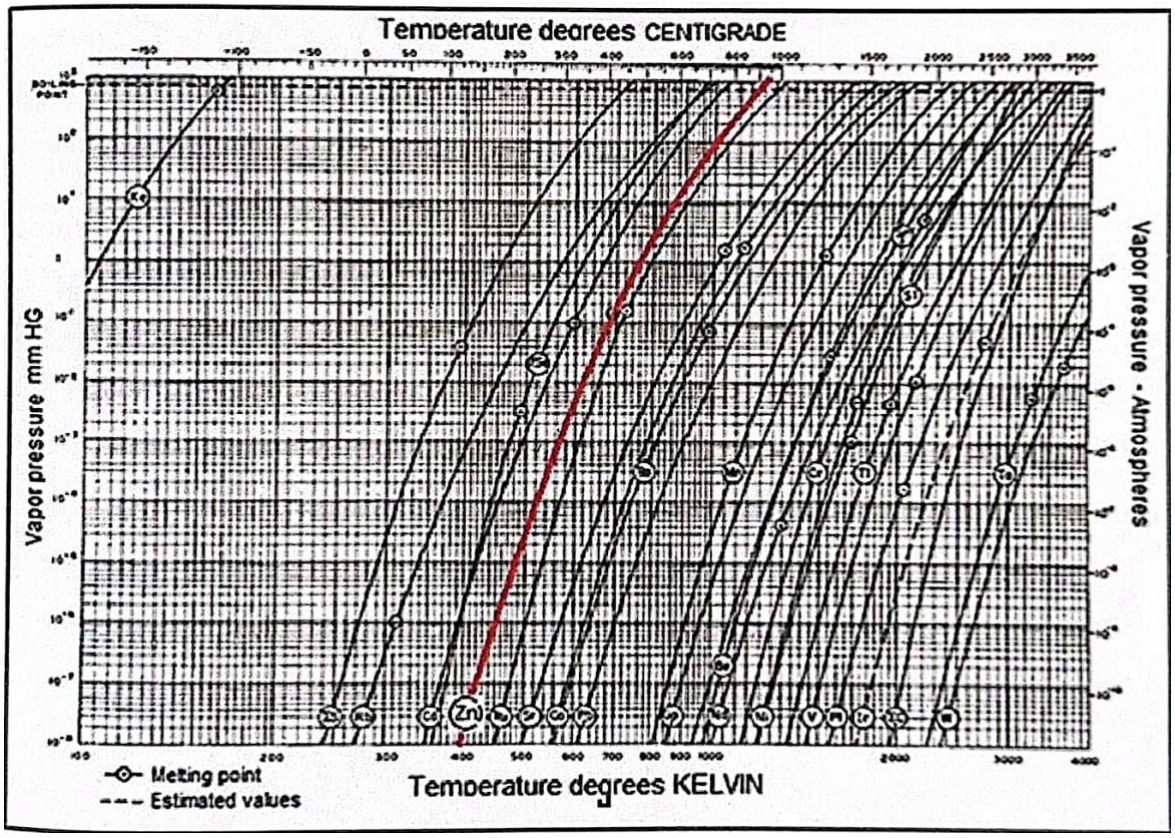


Figure (5-3) vapor pressure of some elements

The porosity also increases dramatically Ascending from sample No. 1 (20%) to sample No. 4 (80%) because the percentage of zinc is greater . As for the density, it decreases for the same reason as shown in the Figs (6-3) and (7-3) .

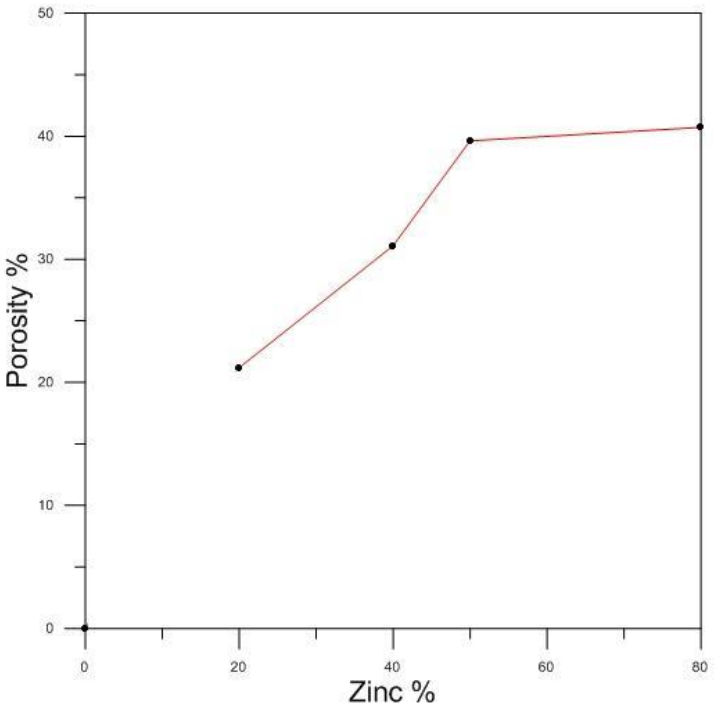


Figure (6-3) relationship between the Zinc% and porosity% after sintering

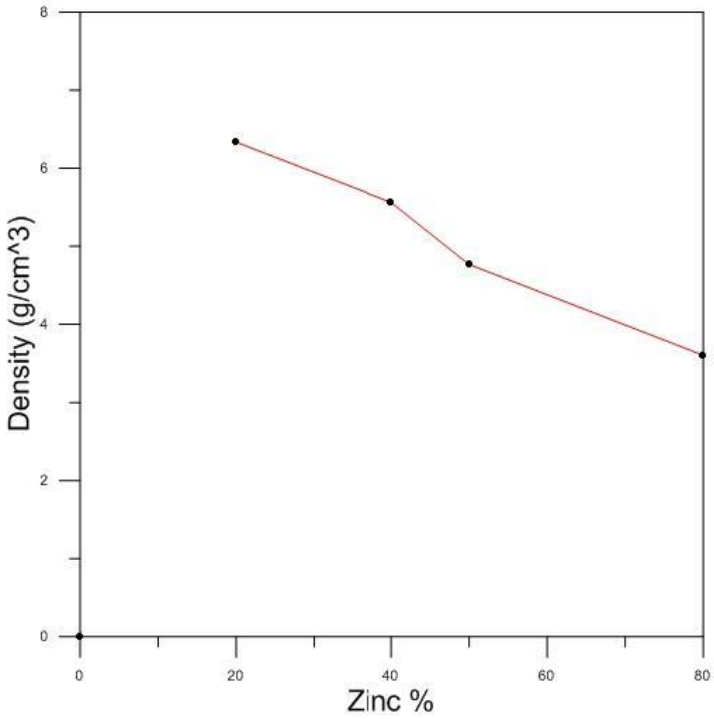


Figure (7-3) relationship between the Zinc% and density after sintering

These conclusions is supported by the microstructure images for samples bellow :

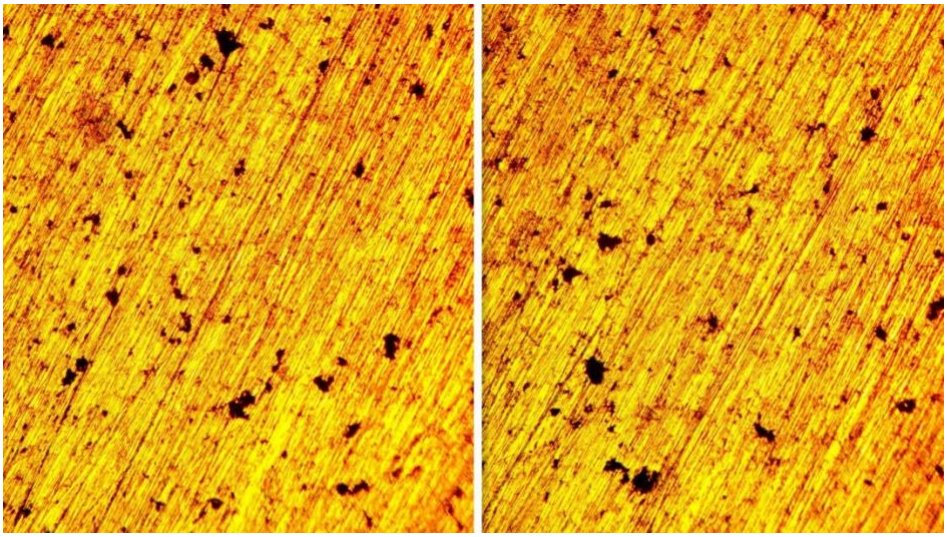


Figure (8-3) Microstructure image showing the porosity in sample 1 (80%Cu 20% Zn)

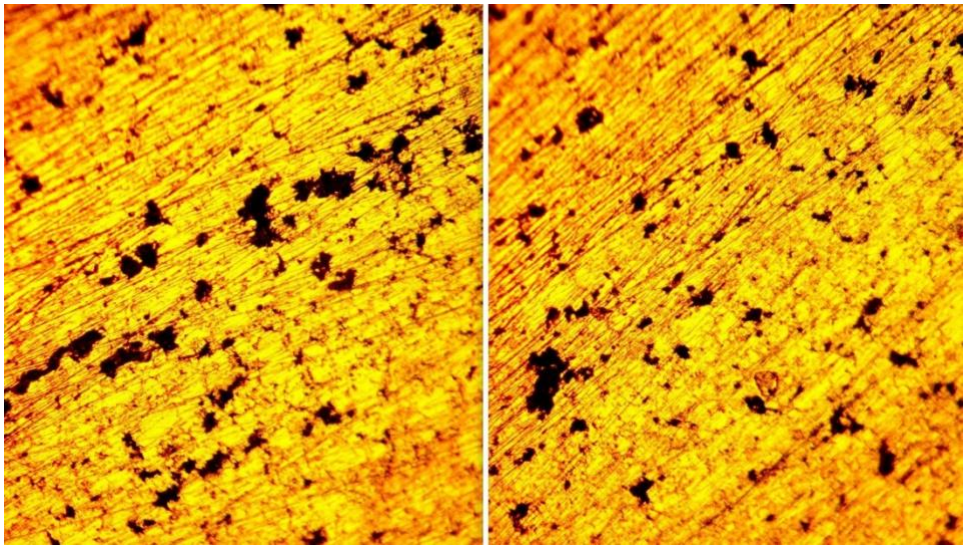


Figure (9-3) Microstructure image showing the porosity in sample 2 (60% Cu 40% Zn)

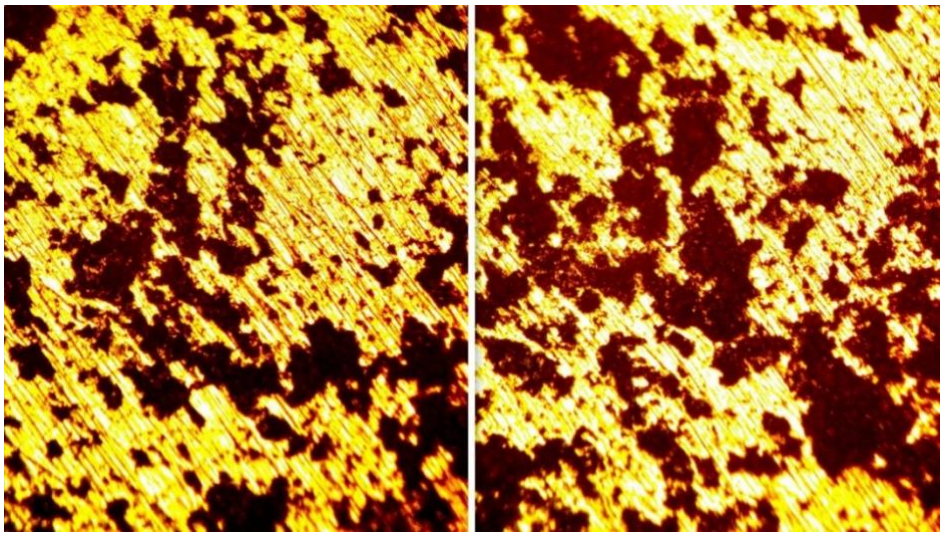


Figure (10-3) microstructure image showing the porosity in sample 3 (50% Cu 50% Zn)

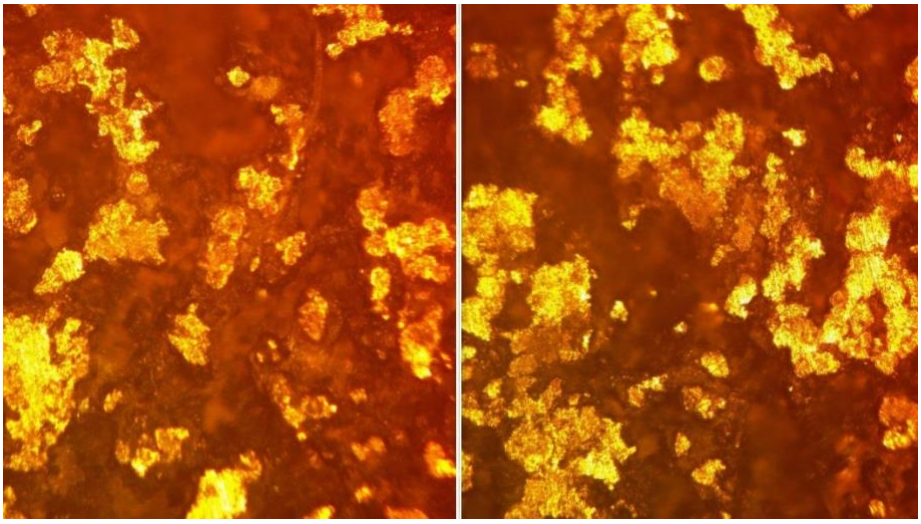


Figure (11-3) microstructure image showing the porosity in sample 4 (20% Cu 80% Zn)

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