

Study of mechanical properties for cold compaction of iron-copper oxide-aluminum oxide composite powder

Abdarazag . A . Hassan Salem. A. Sultan
abdoz_ali@yahoo.comsalemsultan901@gmail.com 0926456362 092 7695525
Khaled. A. Mftah Khaled .E. Alamori
0917309190 0919326910
Centre of Technical research College of Engineering Technology-Janzour

Abstract

In this paper a study has been carried out to produce a new composite material of iron-copper oxide-aluminum oxide by cold compact powder to achieve a significant mechanical property. The first step was used for the preparation of 10 specimens with different weight percentage of the metal powder in cold die where plastic deformation of the powder particles is the major deformation process.

The results show that the highest density of 14.15 g/cm^3 was conducted at specimen number 2 which has percentages of (50% Fe, 40% Cu_2O_3 , and 10% Al_2O_3), also the highest value for compression of 92KN was found in the same specimen.

Finally, the hardness increases with increasing of the weight percentage for copper oxide powder and decreasing of iron powder and percentage range of aluminum oxide powder from 10 to 15 %.

الملخص

في هذه الورقة تم إجراء دراسة لإنتاج مادة مركبة جديدة من أكسيد الحديد والنحاس والألمنيوم بواسطة الضغط على البارد للحصول على خواص ميكانيكية عالية في الخطوة الأولى تم تجهيز عدد 10 عينات بأوزان ونسب مختلفة لمسحوق المعدن في قالب على البارد حيث يعتبر التشوه البلاستيكي لجزيئات المسحوق هو عملية التشوه الرئيسية.

أظهرت النتائج أن أعلى كثافة 14.15 جم / سم^3 تم إجراؤها في العينة رقم 2 والتي تحتوي على نسب (50% Fe، 40% Cu_2O_3 ، 10% Al_2O_3)، كما تم العثور على أعلى قيمة للضغط 92KN في نفس العينة.

أخيراً، تزداد الصلابة بزيادة النسبة المئوية الوزنية لمسحوق أكسيد النحاس وتقليل مسحوق الحديد ومعدل النسبة المئوية لمسحوق أكسيد الألمنيوم من 10 إلى 15%.

1- Introduction

Composite material is a mixture of two or more materials or phases of the same material, insoluble in one another possessing properties which are superior to any of the component materials.

Mechanical properties of materials prepared with such a process are strongly influenced by the characteristics of the powder (particle size and size distribution, particle shape, structure and surface characteristics) and by the way compaction is carried out. Compaction induces very complex states of stress in the powder. Lubrication, powder height in the die, compaction rate and stress triaxiality strongly influence the compaction process and, thus, affect the properties of the products obtained [1]. Accordingly, optimization of mechanical properties of green compacts requires a better knowledge of the relation between powder characteristics and mechanical behavior of the material during compaction (grain sliding and rearrangement, grain deformation). Furthermore, the mechanisms for the development of green strength during compaction are generated by two phenomena, namely (i) particle sliding and inter-locking and (ii) plastic

deformation [2]. The initial stage of compaction leads to rearrangement of the powder from a loose array to close packing. As the pressure increases, the contact area between the grains increases and particles undergo extensive plastic deformation [3]. Green strength has been found to be related to the contact area between particles [3]. Generally, PM studies consider particle size, but neglect morphological parameters of powder grains as they are more complex to define, measure and modify. Grain size can be changed by simple grinding, but the shape of the particles and their surface aspect results from Nano-composites are defined here as a class of materials that contain at least one phase with constituents in the nanometer range ($1 \text{ nm} = (10)^{-9} \text{ m}$). The amount, size and distribution of reinforcing particles in the metal matrix play an important and critical role in enhancing or limiting the overall properties of the composite material. Reinforcing aluminum matrix with much smaller particles, submicron or Nano-sized range, is one of the key factors in producing high-performance composites, which yields improved mechanical properties [4]. There are certain size effects which govern the property of these materials.

2. Experimental procedure

The material which have been used in this study were two ceramics powder copper oxide and aluminum oxide powders with iron metal powders as metal-matrix. About 20 grams of powder (Fe-Cu₂O- Al₂O₃) were placed in a plastic cup which was poured into the die as shown in figure 1. The piston was placed in the die hole and piston was loaded in compression. The compacting pressure was varied and after densities of all the specimens were determined from mass divided (scale $\pm 0.1\text{g}$) by the calculated volume determined from measurements with the dial caliper.



Fig. 1 illustrates the composite powders poured in the die.

2.1 Composite preparation

2.1.1 Powder compact die.

It is decided to use hardened steel (48% HRC) for the punch and die cylinder to withstand the large applied the pressure and abrasion.

Typical die cylinder is shown in figure 2 illustrated the die assemble during the compaction process. By honing the die-wall with a fine stone, the grooves in the surface had depth of less than $0.6 \mu\text{m}$ and $0.8 \mu\text{m}$ at the end of experimentation. After every compaction measurements the die is demounted, and if damage to the die-wall perceptible to the naked eye, the die honed, honing oil removed by rinsing with methyl ethyl ketone (MEK) followed by ultrasonic.

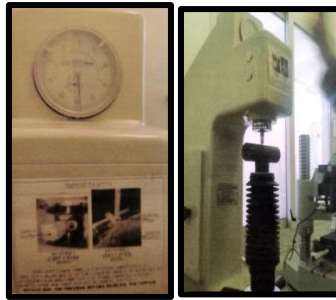


Fig.2 Shows the hardness Of Die.

Cleaned in ethanol and acetone in turn. The clearance between the die-wall and the punches is 0.4 mm in the majority of tests. A hollow cylinder of steel from the inside. 110 mm in length and inner diameter 20mm and 36mm outer diameter as shown in figure 3, and has a high mechanical properties such toughness and hardness to withstand the pressure.

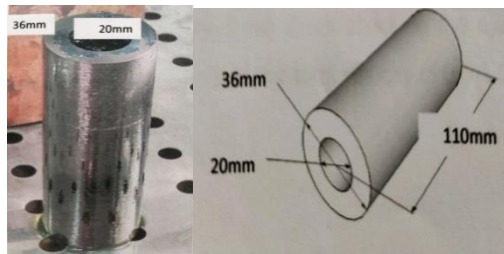


Fig. 3Shows the die Cylinder for powder compact

Results and discussion.

2.1.2 Experimental Program:

Experimental program contains cold compacting the following composite of (Fe-Cu₂O- Al₂O₃) powder are (10) specimens as shown in figure 4, at the same total weight (20gm) for each specimen, as summarized in table 1. Each composite was repeated three times for complete the mechanical properties. The cold compaction pressure for all specimens is constant 150 KN during the work.

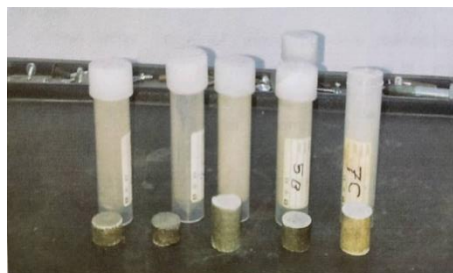


Fig. 4 illustrates the specimen of cold compaction powders of (Fe-Cu₂O-Al₂O₃).

The preparation of the powders, the use of lubricant, the compaction methods and the sintering conditions must be carefully controlled to produce uniform and repeatable porous characteristics. Secondary operations are normally required to enhance metallurgical properties and to allow easier adaptation to the intended application. To produce any specimen from 10 the next steps must be followed.

1. Weight each powder individually to reach the total weight of specimen 20 gm.

2. Manual mixing the three powders in suitable bottle for 15 minutes.
3. The initial stage of metal powders compaction are characterized by plastic deformation of the powders on contact with each other at neck areas. This happens by pouring the mixed powders in the die cylinder assembly, then the compaction pressure applied until 1150 N.
4. The axial force by intron testing machine, axial displacement and the change in pore volume were measured during the test using a distance gauge the relative density was thereby calculated from the volume and mass of the specimen.
5. After compaction the composite (Fe-Cu₂-O- Al₂O₃) powders, the ejection punch use for ejection the compaction powder from the die.
6. The mechanical properties for each specimen will measure by different mechanical and equipment.

There are some pictures in appendix A illustrated the above steps of cold compaction of composite powder (Fe-Cu₂-O- Al₂O₃) summarized in appendix A.

2.1.3 The density of powders compact:

The strength properties of sintered components increase with increasing density but their economy drops with increasing energy input and increasing load on the compacting tool. Thus, it is most desirable, for both economic and technical reasons, to achieve the highest possible compact density at the lowest possible pressure. At first, some definitions are required:

- Specific Weight: $\delta = m/V_T$ (measured in g/cm³), m= mass of the material, V_t= true volume of the material.
- Density: $\rho = m/V_b$ (measured in g/cm³), m= mass of the powder respect compact, V_b = bulk volume (enveloping volume).
- Theoretical Density: ρ_{th} = density of a (practically not attainable) pore-free powder compact (measured in g/cm³).
- Porosity: $\phi = 1 - \rho/\rho_{th}$ (number without dimension).
- Compacting Pressure (die compacting): P = compacting force/face area of compact (measure in N/mm² or MN/m²).

Density-powder contact for above composites curves give information about the frame which a suitable compromise may be found. These curves are generally obtained from standard laboratory tests where a number of compacts are made at different pressure in stainless steel die having a cylindrical bore of 20 mm diameter. The diagram at Figure 5 shows density of powder compact chart for ten commercial powders copper oxide, iron and aluminum oxide and as summarized in Table 1.

Table 1 illustrates the density of 10 specimen of colds compact powder.

Specimen	Density
1	73.12
2	14.15
3	07.49
4	10.61
5	09.79
6	05.79
7	06,36
8	05.54
9	04.54

10	09.80
----	-------

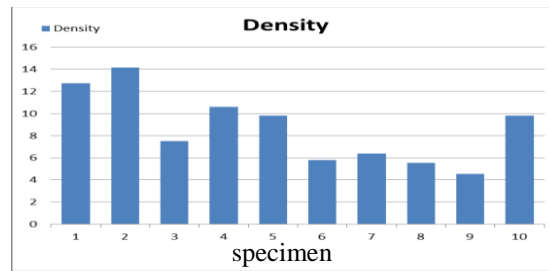


Fig. 5 illustrates the density of 10 specimens of colds compact powder.

It is clear from the above figure that the specimen number 1 and 2 has a high value of density due to the minimum volume of powders compact. That is mean an excellent distribution among three powders at these weight percentage.

4.4 Compression test results:

The compression test done by using universal tension-compression test machine. Eccentric compression test plate was used in all testes in order to prevent shear forces formed by uneven surfaces of the test specimens in Figure 6, test were performed at a cross-head speed of 6m/sec corresponding to a strain rate of $2 \cdot 10^{-3} \text{ s}^{-1}$, respectively, during compression test, the test plates and surfaces of the samples were lubricated in order to reduce the friction between sample and the test plates. At least three tests were conducted for each set of powder samples. The measured strain values were corrected with the compliance of the compression test machine. The result of 10 specimens for different composite cold compact powders are summarized in the next tables from 2 to 6 and Figures from 7 to 12 respectively.

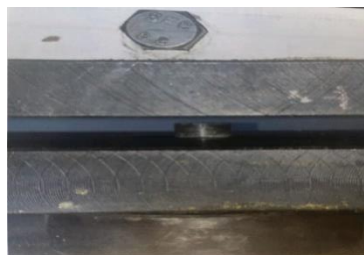


Fig 6 illustrates the specimen during compression test.

Table 2 Shows compression testresult for all specimens:

Specimen	E KN/m ²	Rm N/mm ²	F max(N)	F break (N)
1	3.054	24000	2431	1223
2	4.137	29000	9242.25	6531.2
3	1.458	17500	2755	4812
4	4.107	18000	5654.25	5162
5	5.033	15000	4833.75	4478
6	1.587	80000	2487.50	2019
7	4.949	80000	2566.25	2318
8	5.050	15000	4836.50	3750
9	3.098	50000	1555	936

10	1.635	15000	4726	3500
----	-------	-------	------	------

Table 3 The young's modules (KN/mm²) all specimens of FeCu₂O-Al₂O₃.

Specimen	Density
1	3.054
2	4.137
3	1.458
4	4.107
5	5.033
6	1.587
7	4.949
8	5.050
9	3.098
10	1.635

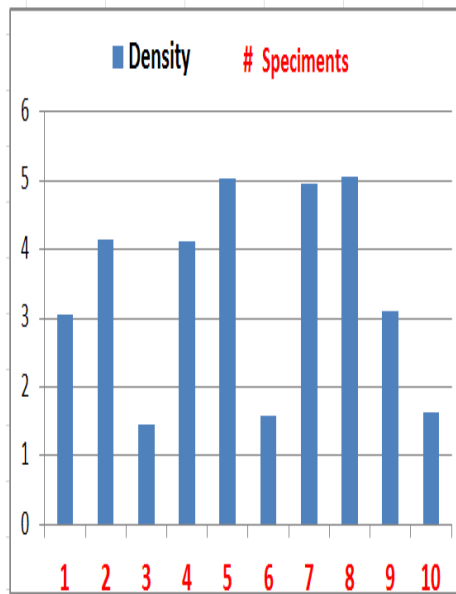


Fig.7 Bar Graph of Young's modules (KN/mm²) for all specimens of (Fe-Cu₂O-Al₂O₃).

Table 4 The bar graph at maximum stress Rm (N/mm²) (Fe-Cu₂O-Al₂O₃) compacted.

Specimen	Maximum Stress Rm (N/m)
1	24000
2	29000
3	17500
4	8000
5	8000
6	8000
7	18000
8	15000
9	15000
10	5000

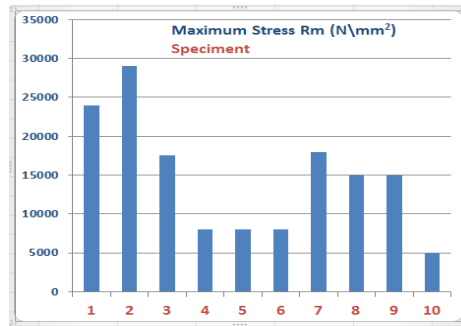


Fig. 8 Shows the bar graph of maximum stress Rm (N/mm²) for all specimens of (Fe-Cu₂O-Al₂O₃)

The Young modules E has a range from 1458 to 5100 N/m² in the results for compression test, the specimen no. 8 (50% Fe – 10% Cu₂O₃ 40% Al₂O₃) have maximum value of Young’s modules because the specimen has high Al₂O₃. While the specimen no. 3(50%Fe-35%Cu₂O-15% Al₂O₃) has a minimum value, this is because the specimen have minimum rate Al₂O₃ percentage. Two specimens no. 1 and no2 have a high value of maximum stress more than (20-29 K/m²). This is due to large amount of copper oxide powder weight percentage which is reached to 40%.

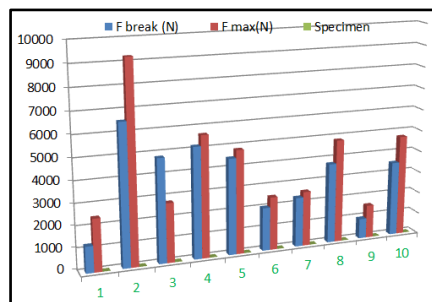


Fig. 9 illustrates the Fmax, N(red bar) and F break, N(blue bar) for all specimens of (Fe-Cu₂O-Al₂O₃) compacted powders.

It is clear from the above table and figure there are a large difference between the maximum force (F max) and break force (F break) except the specimen no 7 and this is due the minimum weight percentage ratio of copper oxide.

4.5 Hardness test:

Rockwell hardness test is most often applied on iron and steel casting where its usefulness is most advantageous as the results represents a sort of average surface hardness because these materials are not uniform on the microscopic scale [7].

It can also be successfully applied to steel bars and plates, and to normalized forgings, that is to forgings which were submitted homogenizing heat treatment, or to fully heat treatment ones. Assuming that the surface is representative of sound metal, for ease of reading the indentation diameters one should have it cleaned from paint, oil or grease, and highly ground with abrasive paper (180 grit).



Fig. 10 Illustrates the micro Hardness testing machine.

Table 5 shows hardness test results for all specimens:

Specimen no	Hardness (HB)
1	23
2	26
3	21
4	18
5	16
6	11
7	8
8	14
9	10.5
10	24



Fig. 11 Illustrates the hardness test result for all specimens of all (Fe-Cu₂O-Al₂O₃) compacted powers.

The Rockwell hardness has a range from 8 to 26 in the result for all specimens. The specimen no.2 (50% Fe- 40% Cu₂O-10% Al₂O₃) and no.10 (40% Fe -50% Cu₂O – 10% Al₂O₃) have maximum value of hardness while the specimen no. (50% Fe- 15% Cu₂O- 35% Al₂O₃) has a minimum values of hardness are due to the high weight percentages of Al₂O₃ and low percentage of Cu₂O of the specimens.

The tensile test strength from diametric compression test is evaluated by substituting the maximum load value in Eq (1), For powder material the tensile strength is a density dependent on the material parameter.

The indirect tensile strength of compact powder Fe. Cu₂O- Al₂O₃ composite with different weight % of Cu were measured. For this purpose Fe – Cu₂O – Al₂O₃ composite of right circular cylindrical shape were fabricated by powder metallurgy process. The tensile stress δ is given by:-

$$\sigma = \frac{2F}{\pi \cdot d \cdot t} \dots \dots \dots (1)$$

Where, F = applied load (N), d = specimen diameter (m), t = Specimen thickness (m).

In this work, it is used the Germany specification [DIN 50 150] tables for measured the tensile strength as represent in appendix A, by use the following equation (2):

$$\sigma \text{ (MPa)} = 3.37 \text{ HB} \dots \dots \dots (2)$$

Where (HB < 175), HB = 0.95HV.

Remember: σ is the tensile strength, HV is the Vickers micro-hardness and HB is the Brinell micro-hardness. [8].

Table 6 Tensile strength results for all specimens.

Specimen no	Average Tensile Strength,MPa
1	212.31
2	219.05
3	208.94
4	205.57
5	202.2
6	192.09
7	188.72
8	198.83
9	192.09
10	215.68

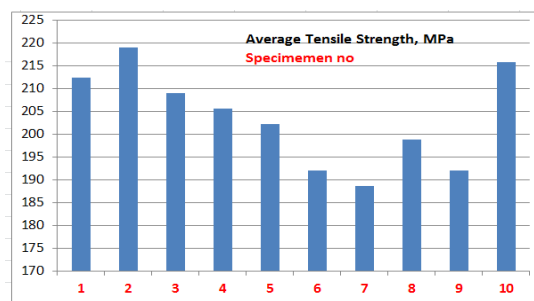


Fig. Bar chart at average Tensile strength (MPa) for all specimens at (Fe-Ca₂O-Al₂O₃) compacted powder.

The Tensile Strength has average of 188.75 to 219.05 MPa in the results for all specimens. The specimen no 2 (50% Fe – 40% Cu₂O – 10% Al₂O₃) and no 10 (40% Fe – 50% Cu₂O – 10% Al₂O₃) have maximum value of tensile strength while the specimen no. 7 (50% Fe – 15% Cu₂O – 35% Al₂O₃) has a lower value. These high values at tensile strength. Due to the high value of hardness for the two specimens.

Conclusions:-

1. The heights value of compact density (P_f)is found 14.15gm/cm³ for composite of specimen no. 2 (50% Fe, 40% Cu₂O, 10% Al₂O₃).
2. With increasing densification, the powder particles are plastically deformed and increasingly deformation strengthened, i.e their yield point is steadily being raised. Simultaneously, the contact areas between particles are increased and, consequently, the effective shearing-stresses

inside the particles are decreasing. Thus, at constant external pressure, decreasing shearing-stresses meet a rising yield point, and all further particle deformation ceases, i.e, the densification process stops.

3. The heights value of compression test is found to be 92 KN for composite of specimen No. 2 (50% Fe, 40% Cu₂O, 10% Al₂O₃).
4. The instantaneous wear rate is a function of Cu₂O content present in the mix and it also effectively controls the mechanical properties in low stress rubbing contact of the composites with harder material. This is due to the fact the wear mostly during this work is of adhesive type and the wear debris formed a third a layer in the contact zone. The presence of hard Cu₂O particulates in the wear debris may have been acted as lubricating pool in otherwise dry sliding contact of the specimen and hardened steel die.
5. The investigation shows that the general behavior may be applied to the mechanical properties as it increases with the increases of Cu content. So it may be said that these results may very well be used to develop a model for prediction of wear behavior of (Fe,Cu₂O-Al₂O₃) powders performs.
6. It is found that the hardness increase with increasing of the weights percentage for copper oxide powder and decreasing of the weights percentage for copper oxide powder with range of aluminum oxide powder from 10 to 15% for the composite powders which have been used.
7. Though all the result were analyses based upon the average readings of more than 30 basic experimentation it has been found that the compression testing machine should also be fitted with more than one transducer that shall ensure constant initial contact pressure for each and every trial.

References:

1. Gaoyang, Y., Huimin, W., Shuhai, C., Lu, W., Jihua, H., Jian, Y., & Zhiyi, Z. (2020). Interfacial reaction between solid Ni and liquid Al in tens of seconds: Dissolution kinetics of solid Ni and formation of intermetallic compounds, *Materials Characterization*, 159, Article 110043. DOI: 10.1016/j.matchar.2019.110043
2. Lai, X., Bach, D., Van, T., Vu, T. H. T., Thang, B. P., Nguyen, H., Vu, N. D. (2019). An investigation on titanium multilayer coatings for enhanced corrosion resistance of carbon steel in simulated seawater by sol–gel dip coating, *Journal of Materials Research and Technology*, 8(6), 6400-6406. DOI: 10.1016/j.jmrt.2019.09.061
3. Wagih, A., & Fathy, A. (2018). Improving compressibility and thermal properties of Al–Al₂O₃ Nanocomposites using Mg particles. *Journal of Materials Science*, 53, 11393-11402. DOI: 10.1007/s10853-018-2422-1
4. .Wagih, A., & Fathy, A. (2017). Experimental investigation and FE simulation of spherical indentation on nano-alumina reinforced copper-matrix composite produced by three different techniques. *Advance Powder Technology*, 28, 1954-1965. DOI: 10.1016/j.appt.2017.05.005.
5. Boltachey G. S. et al. *Acta, Mechanical*, 2009, 204. 37-50p.
6. Biswas K. *Journal of Materials Processing Technology* 2005. 166,107-115p.
7. Bejarano A. et al. *Journal of Materials Processing Technology* 2003. 143-144: 34-50.
8. Gaboriault E. M. The effects of fill-no uniformities on the dandified states of cylindrical green P/M compacts, M.Sc. Thesis Department of mechanical Engineering, Worcester Polytechnic Institute, USA.2003.
9. Lee S. C and Kim K. T. *International Journal of Mechanical Sciences* 2002. 044.1295-1308p.

10. Shima S. and Saleh M.(1993). Advanced in powder metallurgy and Particulate Materials, modeling design and computational methods, Tennessee, USA. 16-19 May.1993. 175-188p.