Physical and Mechanical Characteristics of Heat Treated P/M Parts, Infiltrated By Copper

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Abstract: In this study, the effect of infiltration and heat-hardened treatment and return on physical and mechanical properties of ferrous powder metallurgy products were studied. For this purpose and to prepare ferrous powder and copper powder samples, each powder mixture separately were prepared in a form of a cuboid with floating matrix under tension of 500 MPa dense and crude blocks of iron and copper. For conducting infiltration, a copper sample was placed on each iron sample and this inter-furnace set with reduction atmosphere was sintered in temperature of 1150oC during 30 minutes. A number of samples were austenitized at a temperature of 850 and 925°C for 10 minutes and then after quenching in oil for 2 h were returned at 250°C. Samples were under tensile, hardness measurement, dimensional changes, open porosity and density determination tests. Results show improvement of strength and hardness coincide with increased flexibility in infiltration. Also under the effect of heat treatment, strength and hardness are increased, but flexibility is reduced. Infiltration with copper and heat hardened treatment lead to dimensional changes as fluff in samples.

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1 - Introduction

Condensation is one of essential principles for improving powder metallurgy components quality. Although different methods of compression and sintering are effective to gain this objective, but their efficiency for improving components properties are limit. Usually complete condensation requires applying heat and tension simultaneously to remove pores of a part [1]. In fact, achieving to complete condensation requires process propellant to be sufficient at high temperatures to remove part porosity. A mixture of different parameters such as stress, temperature, additions, strain rate and time could be used for this work [4-2]. For complete condensation of powder metallurgy products, there are several methods such as infiltration, hot forging, powder forging, extrusion, dynamic and explosive powder press. Sometimes applying high stress tension to reach complete condensation is not possible in producing ferrous-powder parts because of high strength of iron powders and size of product, and among above mentioned methods, filtration shows off that require tension to reach complete condensation [6-5].

In infiltration process, pores of a solid part (sintering or crude) will be full with melt alloy or metal. Melting point of a part should be much more than metal or alloy that is in it and therefore the number of systems in which infiltration is used commercially are limited and they limit to parts of iron, molybdenum and tungsten. Despite limitation in number of these systems, produced parts numbers are very much with this method. There are a lot of discussions about theoretical aspects related to powder metallurgy products infiltration in many articles. These discussions have limitations about iron parts infiltration by copper because of copper solution in iron austenite context [7]. The most common method in steel and iron parts is penetration of compressed part of copper powder into crude iron compressed part. This process in which sintering and infiltration are conducted simultaneously is called sintering. In this process with exothermic control rate when products reach to melting point of penetrated material i.e. copper, crude part is sintering sufficiently [8].

About infiltration products, considerable dimensional changes are observed. Increasing Carbon percentage exist in iron part, expansion and growth are reduced because of copper penetration, so that for .2 to 1.5% copper, this dimensions increase reduce to .1% [9]. Because of infiltration process, part porosity and pores are removed and mechanical properties especially toughness is improved, so that for iron part, impact energy increases 3 to 20 j because of infiltration [10]. Also, because of infiltration almost 20% weight of crude part, copper metal is infiltrated. From this amount, about 1.5% copper is as solution and the rest is accumulated as independent phase at the edge of powder particles.

In spite of mechanical properties improvement of iron powder under the effect of infiltration by copper, heat treatment impact also can be considerable on mechanical properties of these products. Evaluating balance diagram (Fe-Cu-C) in different temperatures, it is observed that copper solution in phase is a function of temperature degree. Graphs in figure 1 (temperature isotherms in 850

and 925°C) clearly show this difference. In this study, infiltration effects of iron powder metallurgy infiltration by copper metal and austenitizing temperature in hardening and return treatment on mechanical and physical properties of iron powder metallurgy samples are reviewed.

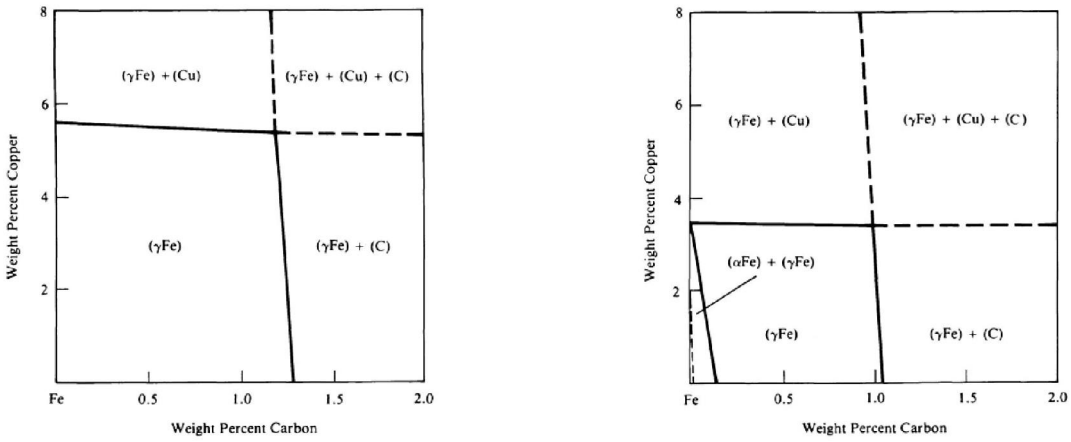

Figure 1 – diagram of three elements of iron - Carbon - Copper, a) temperature of 850°C and b) temperature of 925°C [10].

2 - Materials and method of research

Raw materials including pure iron powder, powder of graphite, copper powder and micro wax are used after physical properties measurement according to table 1. Chemical compounds of iron samples of test were selected according to table 2. After selecting materials, above powders were entered in cone blender and they were mixed for 30 minutes. Circulation speed of blender was 30 rpm.

Table 1. Physical properties of used raw materials

Particle Grading Distribution (μm)	Apparent Density of Powder (g/cm^3)	Powder name
10-160	2/67	Iron
10-63	3/21	Copper
10-63	-	Graphite
10-43	-	Wax

Table 2 – chemical composition of used powder mixture

Pure Iron Wpl200	Wax	Graphite	Powder
others	1	0/8	Weight Percent

After preparing a powder mixture, cube samples with dimensions of $6 \times 12.7 \times 3$ mm and tensile samples of desired powder mixture density were obtained in format of standard ASTM B823 under tension of 500 MPa. Also, several cube sample with dimensions of $3\times 12.7 \times 31.75$ mm were condensed by electrolyte pure copper powder into same frames. Sintering operation of samples under reduction atmosphere (N_2 -%75H_2) was conducted inside continuous strength furnace. Photo spectrometer analysis (quantum) from sintering samples by Foundry Master UV was conducted to determine chemical composition of under study alloys. Conducting infiltration treatment of ferrous powder parts by copper, during sintering treatment one copper sample was placed on any iron sample. Sintering temperature of 1150°C and time of heating in this temperature was 30 min. A number of infiltrated samples were under heat treatment. Samples in two temperatures of 850 and 925°C were austenitized for 10 minutes at salt bathroom (50% $NaCO_3$ - 50% NaCl) and then they quenched at Oil. Returning sample was done at 250°C for 2 hours.

Various samples of coding system in table 3 were used for study and easy reporting.

Table 3 -Coding system of different samples

Sample Properties	Sample Code
Raw Iron Sample	A
Sintered Sample	B
Sintered and Copper-Infiltrated Sample	C
Sintered, Infiltrated, Hardened, and Tempered Sample (Austempered in 850 ºC for 10 minutes, Quenched in oil, and Tempered in 250 ºC for 2 hours)	D
Sintered, Infiltrated, Hardened, and Tempered Sample (Austempered in 925 ºC for 10 minutes, Quenched in oil, and Tempered in 250 ºC for 2 hours (	E

All sintered samples were under density and porosity measurement test according to Standard ASTM C373-72. Dimensions of raw and sintering samples were measured under different conditions (infiltrated and non-infiltrated) by digital caliper with accuracy of 0.01 mm and dimensional changes were calculated relative to frame. Samples condensation amount was measured and reported according to equation 1.

$$\varphi = \frac{\rho_S - \rho_g}{\rho_t - \rho_g} \times 100 \quad \text{Equation (1)}$$

Where, $_S$ is sample density after sintering, $_g$ is raw sample density and $_t$ is sample theoretical density.

Tension test was conducted on sintered iron tension samples, iron infiltration with copper (in non-heated and heated treatment conditions). Hardness test was done on all cube samples by Brinell method by sinking ball in diameter 2.5 mm and applied load of 187.5. Cube samples in sintering conditions and sintering and infiltration by copper (in non-heated and heated treatment conditions) using conventional metallographic methods were reviewed micro structurally.

3 - Results and discussion

3-1 - Chemical composition and microscopic structure

Chemical composition of under study powder samples resulted from quantum test is given in table 4.Figure 2- (a), microscopic structure of sintered iron sample is shown. As it can be seen, structure is composed of perlite and a little of ferrite. Despite high carbon content of samples, the existence of ferrite phase is because of decarbonized atmosphere (oxidant) while sintering. Despite of using decomposition ammonia gas (a mixture of hydrogen and nitrogen) due to high dew point in input gas mixture entering furnace, decarbonized operation is done according to equation (2) [3].

Equation (2)	$Fe + C + H_2O \leftrightarrow Fe + CO + H_2$

Figure 2 - (b) structure of infiltrated iron sample is shown. So that as it can be seen copper penetrate in pores of iron sample and microscopic structure is fully perlite. The reason of removing ferrite phase related to copper solubility up to 2% in austenite that during cooling makes eutectoid transition for less carbon and makes structure fully perlite [11].

Table 4 - Chemical composition of under study alloys

Element Name	Weigh Percent
Fe	99/22
C	0/67
Mn	0/03
Si	0/08

Figure 2 - (c), microstructure of infiltrated metal sample in 850°C austenitized, quenching in oil and returned at 250°C is shown. Structure consists of perlite with small martensite phase. Under the effect of austenitization temperature, volume percentage of martensite phase is increased in this field, so that a large plate martensite in a field of austenite is its result. This is evident in figure 2-(d). In this figure, the existence of rest austenite whose a large part of that is remained after return, can be related to higher solubility of copper (up 6 percent) (Figure 1). In infiltrated samples under the effect of solubility of copper in austenite, strength of austenite was increased because of solidification solution and thus M_s and M_f temperatures are reduced. Under the effect of increasing shear strength of austenite containing copper and low temperature of martensitic transformation, driving force of changing austenite to martensite transformation is increased and it causes to existence of remained austenite phase at room temperature and after returning [9,1].

3-2 - Physical properties

Raw density samples were calculated by measuring mass and determining sample dimensions with geometric method that is 6.35 gr/cm^3. As it can be seen in table 5, by sintering at 1150°C for 30 min, density of samples were increased to 6.45 gr/cm^3. According to equation 1, the amount of condensate percent (φ) computed in sintering samples is 7 percent in this mode. Under the effect of infiltration operation with copper and in sample C, samples density were increased from 6.45 to 7.62 gr/cm^3. The

reason may be related to penetration of copper metal in open porosities in sample during sintering process [9]. In this mode, the amount of condensation reaches to 77 percent. Under the effect of hardness heat treatment in sample D, sample density does not change. The reason is small volume change resulted from transformation of austenite to martensite. Before heat treatment, sample structure includes perlite and copper metal that under the effect of heat treatment because of low austenitization temperature, martensite phase amount obtained is small that result in no change in density of heat treatment sample [6]. Final structure of sample E consists of martensite and residual austenite and copper. Because of dimensional expansion under the effect of martensite formation, density of samples is reduced to 7.60 gr/cm^3 and condensation percent in this sample is about 76 percent.

Table 5 - Physical properties of different samples

Open Porosity (%)	Density (g/cm^3)	Sample Name
17	6/35	A
15	6/45	B
-	7/62	C
-	7/62	D
-	7/60	E

Results of sample dimensional changes measurements under different conditions are given in Table 6. As it can be seen, dimensional changes in sample A is spring back. Under the effect of sintering process of crude iron sample at 1150^{o}C, dimensional changes are considered as reduction size or shrinkage. The contraction rate of this mode is approximately .2 percent. Under the effect of filtration by copper (sample C) due to intrusion of molten copper among ferrous powder particles under capillary force effect, some fluff was occurred [9]. In sample D, despite doing heat treatment due to low austenitizing temperature, a little percent of martensite phase is formed that does not cause many dimensional changes but in sample E, due to high austenitizing temperature (925^{o}C) and high percentage of martensite phase [11] dimensional changes are relatively considerable.

Table 6 - Dimensional changes of different samples

Swelling (%)	Contraction (%)	Rebound (%)	Sample Name
-	-	0/1	A
-	0/2	-	B
1/75	-	-	C
1/97	-	-	D
2/91	-	-	E

3-3 - Mechanical properties

Due to tensile and hardness test results that are given in table 7, it is clear that infiltrated operation on ferrous products was led to improved tensile properties. As it is evident, sample C compared with sample B has high yield strength, tensile strength, ductility and higher toughness. This is partly because of loss porosity in filtrated samples with Cu atoms penetration into porous that is itself act as crack [6]. Also under the effect of infiltration of samples of iron by copper metal, perlite structure appears that this causes to high strength of sample compared sintering samples without infiltration. [2].

Because of hardness and returning heat treatment, tensile and strength yield of samples are increased due to martensite phase formation. The amount of this phase in sample E compared with sample D is higher due to high austenitization temperature. Heat treatment doing cause to flexibility reduction, so that in non-heated treatment, elongation amount is about 6.1 percent, and this parameter is reduced in heat treated sample to about 3.1 percent.

Table 7 – Mechanical properties of different samples

Hardness (BHN)	Elongation (%)	Yield Strength (MPa)	Tensile Strength (MPa)	Sample Name
67	3/2	152	210	B
150	6/1	565	697	C
257	4/2	648	802	D
340	3/1	702	855	E

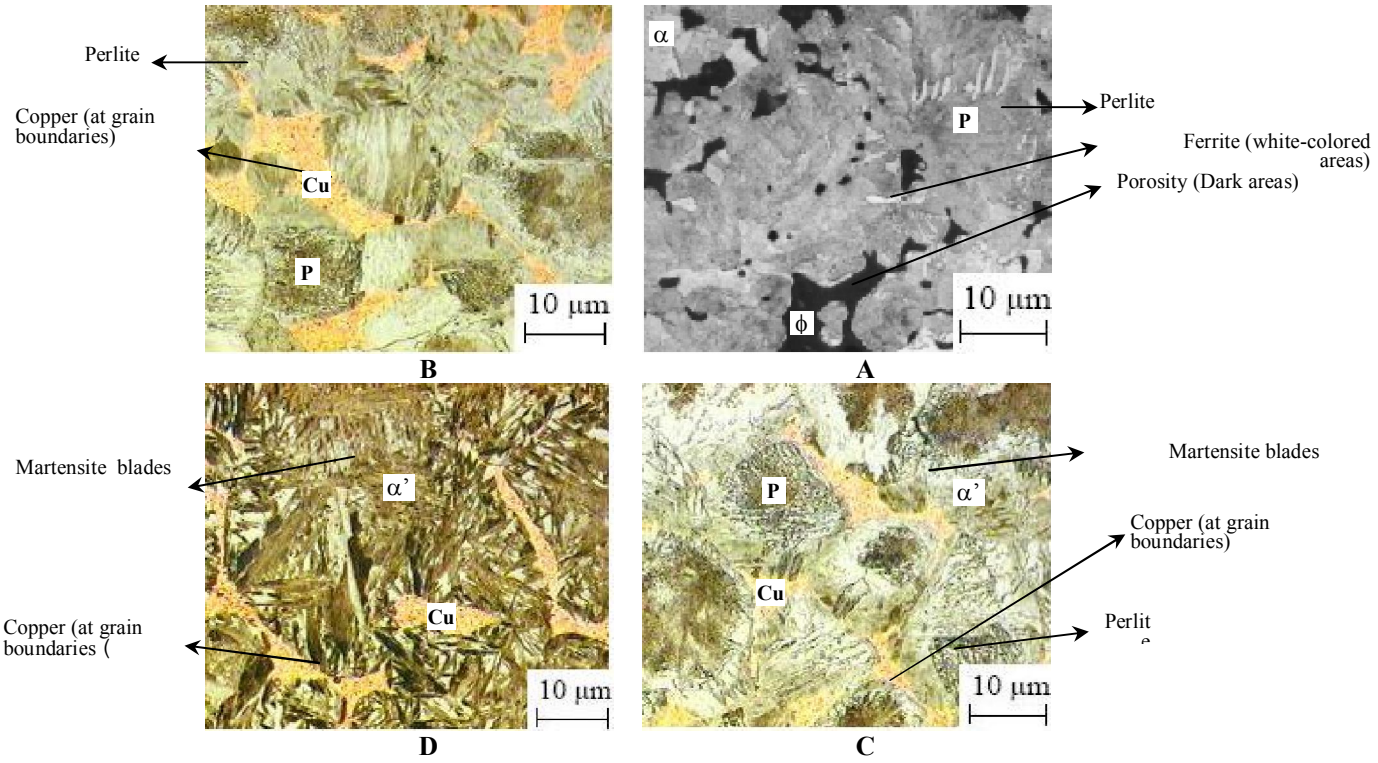

Figure 2 - Microstructure of iron samples produced by powder metallurgy method: a) sintering (containing 15% porosity), b) sintering and infiltrated with copper, C) sintering and infiltrated with copper, austenitized at 850°C, quenching in oil, returned at 250°C, and D) sintering and infiltrated with copper, austenitized at 925°C, quenching in oil, and returned at 250°C

4- Conclusion

1) – Infiltrating ferrous powder metallurgy parts with copper is the most effective and easiest method to achieve complete condensation in these products if samples condensation reaches 77% in this study.

2) - Infiltrating ferrous powder metallurgy parts with copper causes to increase strength of 210 MPa to 697 MPa and hardness increase from 67 BHN to 150 BHN. Also, final elongation percent is increased from 3.1% to 6.1%. This is due to lower porosity of about 15% to zero.

3) – Hardness and return operation cause to increase strength of infiltrated sample from 697 MPa to 802 MPa and 855 MPa in austenitizing temperature of 850 C and 925 C, respectively.

4) - Increasing austenitizing temperature in heat treatment increases the amount of copper dissolved in austenite and cause to increase volume percent of martensite and hardness and strength increase and flexibility reduction of products after hardness treatment.

5) – In infiltrating operation of ferrous powder metallurgy products by copper, dimensional changes about 1.75% is observed.

6) - The amount of samples fluff with increasing austenitizing temperature from 850 C to 925 C of 1.97% to 2.91% amount is increased due to volume percent increase of martensite phase.

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