Changes of Fe matrix's lattice constant during liquid phase sintering of Fe-Cu-C compacts by x-ray diffraction techniques

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ABSTRACT The dissolution of graphite and copper during sintering of PM steels prepared from iron, copper and graphite powder mixes were studied using X-Ray Diffraction method. This paper present the investigation carried out to study the changes of iron's lattice constant during liquid phase sintering of the compacts. The electrical conductivity measurement method was also used for determining the extent of carbon and copper dissolution and its influence on the formation of sintered compacts. In the experiment, the Fe-Cu-C powders were compacted into a pellets using hand press machine and were then sintered in a 5% H_2 + 95% N_2 gas atmosphere at different sintering temperature in the range of 400 °C and 1200 °C. The effect of sintering parameters on the mechanical properties of the sintered compacts was studied to find a correlation between mechanical behaviour, microstructure, and the resistivity in order to develop non-destructive testing method. It was observed that measurement of Fe matrix's lattice constant and electrical conductivity of sintered compacts could be a viable method in studying all stages of sintering process.

(Lattice constant, Electrical conductivity, Liquid phase sintering, Microstructure)

INTRODUCTION

In the manufacturing of sintered steels parts, carbon is the most effective alloying elements additive because of its manifold reciprocal effects on iron and other alloying element. For technological and economical reasons this inexpensive material is introduced in the form of graphite powder. Therefore, knowledge of the dissolution behaviour of carbon in the iron matrix during sintering is importance for quality assurance [1-2].

Surprisingly, the mechanism, despite its technical importance, is not well understood. Another approach for studying the carbon dissolution mechanism and the influence of manufacturing condition was regarded necessary [3]. Electrical conductivity measurement could be an interesting method for evaluating possible variation in material structure occurring during processing of sintered parts [4].

The use of X-Ray Diffraction method in the study of the dissolution of graphite and copper in iron matrix during sintering of PM steels prepared from iron, copper and graphite mix has not yet been reported by other investigators. The purpose of this work is therefore to discuss the applicability of this technique for determining the dissolution of copper and graphite in iron matrix and the electrical for conductivity measurement as a reliable method for determining the changes in material structure occurring during sintering of the compacts.

MATERIALS AND METHOD

Raw Material

A commercial grade powders used for this study were iron, copper and carbon. Zinc Stearate was used as lubricant to provide effective lubrication between powder particles, uniform pressure distribution during compaction and to facilitate ejection part from the die after pressing. The powders were characterised for pycnometric density measurement and particle size as tabulated in Table 1. Pycnometry density was

determined using pycnodensity meter and particle size distribution was carried out using sieve method analysis.

Table 1: Powder characteristic.

	Iron	Copper	Carbon
Pycnometry density (gcm^{-1})	7.86	9.68	2.02
Particle size (μm)	Fe < 63	Cu < 38	C < 38

Powder Mixing

A 500g blend containing iron, copper and carbon of mass percentage ratio 96:3: 1 together with 0.75 mass % zinc stearate were loaded in 200 ml polypropylene container. The blended powders were mixed using double cone mixer at a mixing rate of 60 rpm for 60 minutes to homogeneously blend the constituents.

Powder Compaction

The optimum compaction force for establishing the optimum green density of the specimen was established. The determined optimum compaction force of 7000 kgf was used throughout the investigation to produce five pellets. The homogeneously blended powders were then compacted into pellets using a hand press machine.

Differential Thermal Analysis

Prior to sintering, thermal analysis of the compact are performed by using Netzch STA differential analyser. The thermal analysis behaviour of the compact was investigated in the range of 25 °C to 1590 °C under a dynamic nitrogen gas of 10 ml/min. The heating rate used was 10 °C/min.

Sintering Process

The sintering process were conducted in a batch furnace with heating rate of 10 ° C / min. The specimens were placed on ceramic plate and kept at the centre of the furnace. The sintering temperature was varied between 400 °C to 1200 °C to determine the effect of sintering temperature upon properties of compacted samples. Sintering was carried out in the nitrogen-hydrogen atmosphere containing 5% hydrogen.

X-Ray Diffraction Analysis

The dissolution effect of graphite and copper in the iron matrix was evaluated using x-ray diffraction analysis on a Bruker diffractometer using Cu Kα radiation.

Characterization of sintered body

The linear shrinkage of the sintered specimen was determined by comparing the difference in the diameter of the green body and sintered body. The electrical resistivity of the compacts was examined at room temperature after sintering using a Thompson bridge. The density of sintered body was measured by the water displacement method. The electrical conductivity of sintered compacts was examined after sintering at room temperature. The microstructure was observed under optical microscopy and the hardness of the sintered parts was determined using Rockwell macrohardness tester.

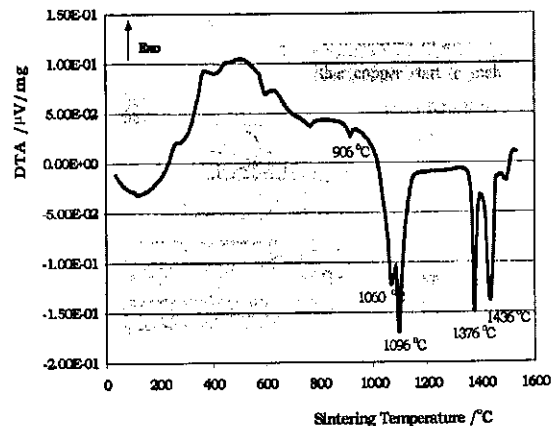## RESULTS AND DISCUSSION

Differential Thermal Analysis

Figure 1 : DTA curve of the mixed raw materials in Nitrogen gas

Figure 1 shows the differential thermal analysis profile for the mixed raw materials. Results show that at 906 °C the transformation of alpha (α) iron to gamma iron (γ) occurred. The melting of copper start at 1096 °C as indicated by presence of sharp endothermic peak and start to dissolve in the iron matrix.

Physical and Mechanical properties

The linear measurements of compacts sintered at. 400 °C to 900 °C in Table 2 showed no shrinkage from the die size which is primarily due to the fact that no formation of the grain boundary in the compacts.

Table 2: Physical properties of the mixed raw materials compacted at 7000 kgf and sintered in 5% hydrogen in nitrogen

Temp /°C	Density / gcm^{-3}	Dimensional Change / %	Hardness / HR_B
400	6.76	0.00	64
500	6.78	0.00	66
600	6.79	0.00	66
700	6.78	0.00	66
800	6.78	0.00	66
900	6.80	0.00	68
1000	6.90	+0.61	70
1100	7.00	+0.46	74
1200	7.01	+0.45	78

However for the compact sintered at 1000 °C a growth of about 0.53% from the die size is noted which is primarily due to the diffusion of carbon in the iron matrix. A further increase in sintering temperature to 1100 °C caused an increase in linear growth to about 0.61 % (see Table 2). This increase in growth is due to the fact that copper start to melt and dissolve in iron matrix. A further increase in sintering temperature to 1200 °C caused the growth to decrease to 0.46%. This is due to near complete particle to particle bonding. The hardness value increased from 68 to 78 HR_B by increasing the sintering temperature from 900 °c to 1200 °C. This increase is related to near complete particle to particle bonding.

Electrical Resistivity

The change of electrical resistivity of the compacts at varying sintering temperature is illustrated in Figure 3. The resistivity of the compacts decreases as sintering temperature increases due to the removal of insulating lubricant layers between particles. It can be assumed that the drop in resistivity of the compacts is caused by the formation of sintered contacts, which enhances the conductivity of the material. The transition of the matrix's crystal structure from body centred cubic (bcc) to face centred cubic (fcc) could also play a role in the increase of the conductivity behaviour of the compact after the alpha (α) to gamma (γ) phase transition in the iron matrix.
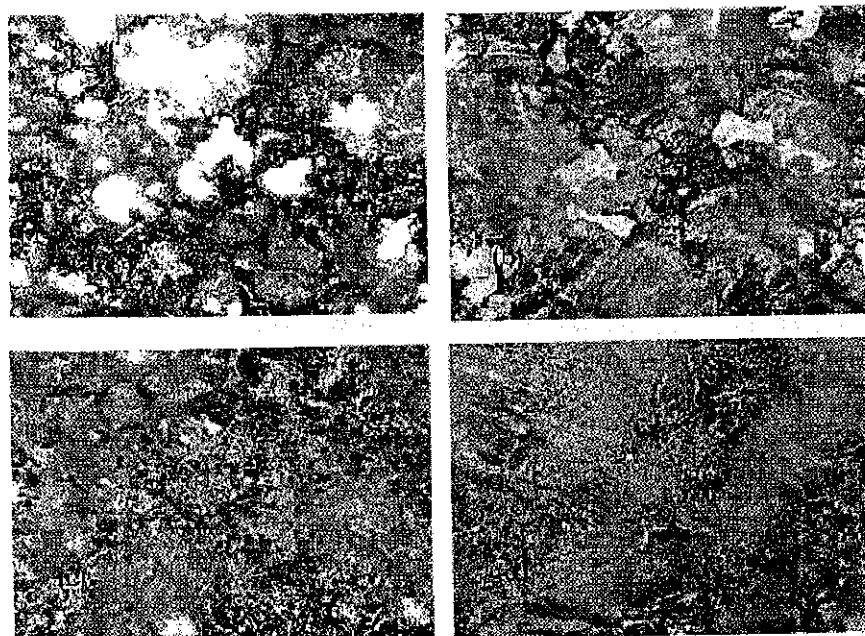

Figure 2. Photomicrographs of compacts sintered at (a) 900 °C (b) 1000 °C (c) 1100 °C (d) 1200 °C
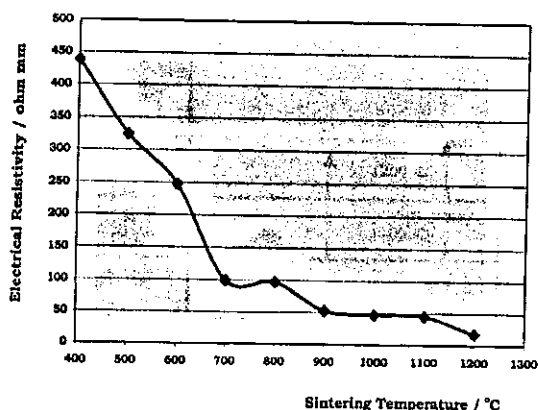
Figure 3. Electrical Resistivity of the compacts as function of sintering temperature

X-Ray Diffraction Analysis

The influence of sintering temperature on the phase structure of the compacts as presented in the XRD spectrum (Figure 4) is clearly visible as peak of carbon and copper start to disappear at 1100 °C. The diffusion of carbon is completed before copper is fully diffused into iron matrix.

Analysis of the XRD spectrum indicates that the 'd' value representating lattice spacing 'a' of iron matrix changes with increasing temperature. The calculated lattice constant, a, for iron was calculated from Fe(110) plane. The changes in lattice constant values (a), for iron with increasing sintering temperature was plotted as a function of sintering temperature and is shown in Figure 5. Result indicate that the 'a' values for Fe (110) plane decrease from 400 °C to 600 °C which could be due to the stress relieving and recrystalisation process of the compact. The increase in 'a' value at 700 °C is caused by the grain growth of iron in the alpha (α) phase and the diffusion of carbon in the iron matrix. Sintering in the (α) phase of the compact shows gradually decrease in lattice constant.

During the α→ γ phase transformation (i.e at round 908 °C), as noted in the differential thermal analysis profile, the lattice constant show an increase value at 1000 °C which could be probably due to this transformation and the phenomena of copper begin to melt and diffuse in iron matrix. The decrease in lattice constant after the transformation of the α→ γ phase probably show that the diffusion of the carbon and copper completed at 1100 °C.
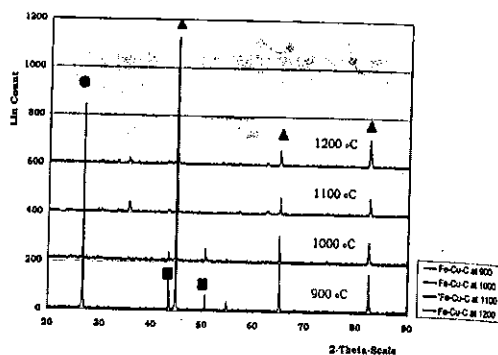

Figure 4. The XRD pattern for the compacts sintered at different sintering temperature. (Key: ▲= Fe: ●= C: ■= Cu)
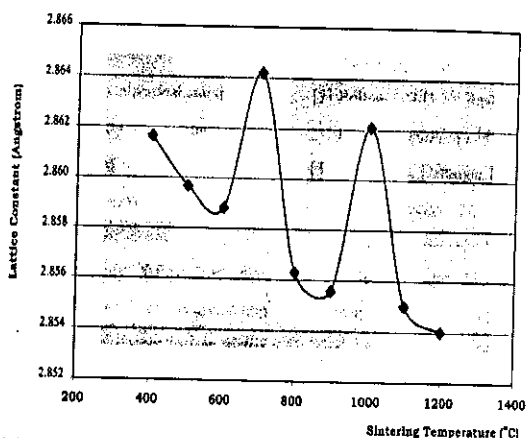

Figure 5. Lattice constant of Fe as a function of sintering temperature

CONCLUSION

1. In the present study, the results show that the sintering temperature is a critical factor influencing the sintered microstructure, densification behaviour and hence the hardness of the Fe-Cu-C materials.

2. Measuring the electrical conductivity of sintered compacts could be a reliable method in studying all stages of sintering process.

3. The applicability X-Ray Diffraction method in the study of the dissolution of graphite and copper in iron matrix during sintering of PM steels prepared from iron, copper and graphite mix is still at its preliminary stage and more work need to be carried out in order to establish more conclusive evident.

Technology and the Environment of Malaysia through IRPA project No. 30-01-01-2001.

REFERENCES

1. Mazli Mustapha, Sarnsiah Sulaiman, Siam Satimin and Hamdan Mohamed. (1999) *Malaysian Science & Technology Congress:* 84-91.

2. Bolton, J .D., Baah, H.O.(1999) *Powder Metallurgy* **34** : 273-279.

3. Simchi, A., Danniger H, and Gierl, c. (2002) *Powder Metallurgy* **44** (2) : 148-156.

4. MPIF , "Powder Metallurgy Design Manual", 1998, Princeton, N.J.