

# Electroless Nickel Plating of Iron Powders

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*This work illustrates the technical feasibility of the electroless nickel plating of iron powders. The coated powders exhibit a high level of homogeneity and the sintered compacts have good mechanical properties. Decreasing powder size increases the nickel concentration and improves diffusion by particle-to-particle contact. Alloying is promoted by reducing diffusion distances.*

## INTRODUCTION

Electroless nickel is described<sup>1</sup> as a nickel-phosphorus alloy which can be applied to metallic surfaces as well as semiconducting materials to provide coatings of a uniform thickness and with excellent chemical and physical properties. The term "electroless" is applied to these coatings since they are deposited without the electric current used in conventional electroplating. While electroless nickel plating has been extensively reviewed by Spencer,<sup>2</sup> various other works have also explored the area.<sup>3-7</sup>

The production of sintered, low-alloy iron/steel from mixtures of the elemental powders may not produce a chemically homogeneous product under normal sintering time and temperature conditions. Consequently, heterogeneity in the material's microstructure results in inferior mechanical properties.<sup>8-9</sup> The presence of porosity is the major factor in this condition, but there is also some evidence that the mixing/blending of elemental powders results in the heterogeneity of the compacts. With pre-alloyed powder particles, the problems of heterogeneity are largely eliminated, but these powders are very hard. Their compressibility becomes so low that the necessary plastic deformation for interfacial contact within the sintered mass for effective diffusion is difficult to obtain at normal compaction pressures. Excessive residual porosity can result.

Composite (coated) powders that improve homogeneity while retaining the good compressibility of elemental powders have been considered to alleviate the problems. The present work looks at the feasibility of nickel coatings for iron powders by an electroless plating method using a mechanical stirring technique.

## THE PROCESS

Electroless nickel coating produces an amorphous deposit in the as-plated condition. Since the deposit is not dependent on current distribution, it is almost uniform in thickness (Table I), regardless of the size or shape of the coated or plated surface. The process is a cementation reaction represented by



Although the reaction may occur slowly, it is theoretically possible to cement nickel from its solution onto an iron substrate. The iron powder particles provide a large surface area for chemical reaction, resulting in effervescence and foam formation on the solution surface caused by hydrogen gas evolution. However, this frothing action results in the loss of iron powder particles from the plating solution container. The nickel plating solution also loses its greenish color substantially after the first few minutes of plating, indicating that the reaction proceeds more quickly within the first few minutes than during the rest of the plating time.

The coated powder itself does not show any appreciable change in color, becoming dull and oxidizing slightly when dried in an electric oven. Oxidation may be a result of the inability of the nickel coating to form a strong, non-porous protective layer on the surface of the iron particles.

## PARTICLE SIZE DISTRIBUTION

Figure 1 clearly shows that the green compact density decreases with decreasing particle size. This effect might result from the fact that increasing the particle size leads to increase in apparent density by promoting particle movement and a more desirable distribution of stresses within the powder mass during compaction. Greater deformation—and hence increased densification—results. Figure 2 shows that porosity increases with decreasing particle size. This relationship could be explained by the compacts produced from the powder fraction of smaller particle size ranges which have a greater number of cavities or voids.

The results given in Figure 3 show that at a constant compaction pressure, sintered density increases with decreasing particle size. This phenomenon might indicate that there is a greater driving force for sintering with decreasing particle size due to more pore-solid interfacial area in the sinter mass. With a smaller size, there is probably greater interparticle contact, providing more paths for

Table I: Coating Thickness of the Powder Particles

Specimen	Average Thickness ( $\mu\text{m}$ )
100 mesh	4.030
150 mesh	4.039
200 mesh	4.028
325 mesh	4.049

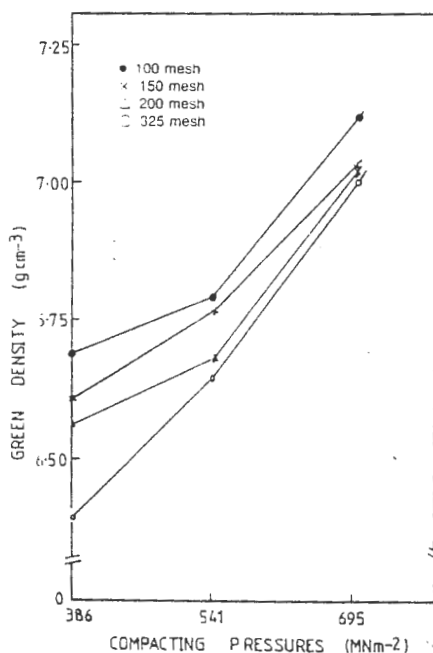


Figure 1. The effect of particle size distribution on green density at different compacting pressures.

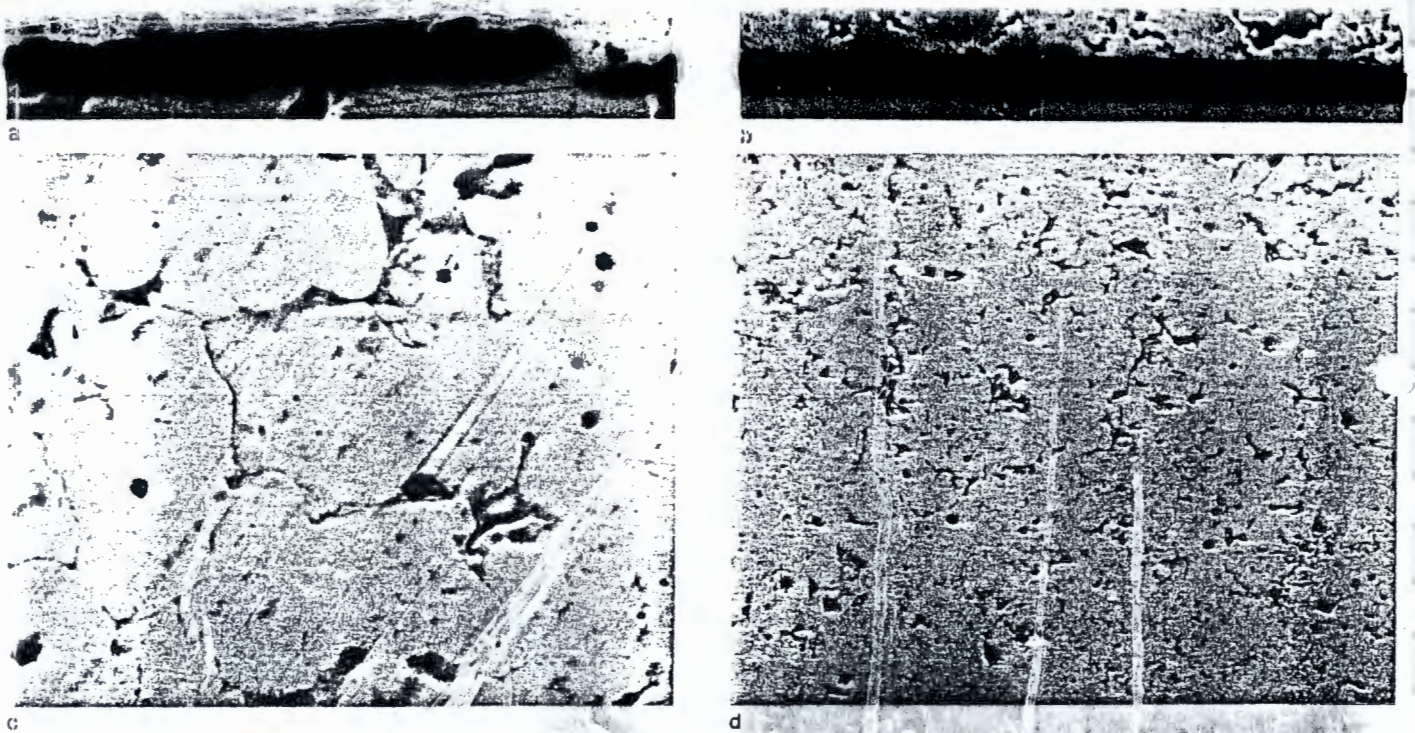


Figure 2. Sintered at 1120°C, these compacts (1000×) at 695 MNm<sup>-2</sup> were etched in a two percent nital reagent. (a) 100 mesh—mass (M)(gm): 59.53; volume (cm<sup>3</sup>): 20; density (gm/cm<sup>3</sup>): 2.976; sieve size (μ): 150; mean particle diameter (μ): 0.1633; μm: 163; and specific surface area (cm<sup>2</sup>/gm): 123.52. (b) 150 mesh—mass: 56.49; vol.: 20;

den.: 2.82; sieve: 105; dia.: 0.122; μm: 122; and surface: 174.40. (c) 200 mesh—mass: 56.02; vol.: 20; den.: 2.80; sieve: 75; dia.: 0.0902; μm: 90; and surface: 238.10. (d) 325 mesh—mass: 55.16; vol.: 20; den.: 2.76; sieve: 31; dia.: 0.0502; μm: 50; and surface: 434.78.

#### EXPERIMENTAL PROCEDURES

Four different fractions of the Hoganaes iron powder ASC 100.29 were made using 100, 150, 200 and 325 mesh sieves. The employed commercial nickel plating solution was diluted with distilled water in the proportion of 50:50. The composition consisted of nickel sulfamate (500 ml/L), acetic acid (30 g/L), and nickel chloride (5 g/L). A pH of 4.8 was maintained by adding 10% sulfuric acid or 50% sodium hydroxide solution with slow stirring. A temperature of 90°C was used throughout for the experiment.

Several platings of one hour each were made for the different fractions of the iron powder. A mechanical stirrer was used to bring the iron particle additions into suspension in the so-

lution. The coated iron powder, after being repeatedly washed with distilled water and methanol, was dried in an electric oven with an air atmosphere at 120°C. The coated powder was tested for nickel using dimethyl glyoxime dissolved in methanol. A purple color precipitate revealed the presence of nickel. Microprobe analysis was also used to spectrally analyze the coated powders in both the green and sintered states.

Tensile specimens (green and sintered) were made from the coated powders using tensile specimen dies with 6.45 sq. cm. surface areas. A Denison hydraulic press was used to make compacts of 386, 541 and 695 MNm<sup>-2</sup> pressure for each of the four different powder particle size ranges.

The compacted specimens were sintered for one hour at 1120, 1080 and 1050°C using an atmosphere of cracked ammonia at 2.27 m<sup>3</sup>/h and a furnace conveyor speed of 3.81 cm/min. The green and sintered densities of the samples were determined by calculations, and the green and sintered strength was determined using an Instron tensometer. The percentage nickel content of the compacts was determined using an SEM by analyzing the bulk mounted sample. A micrometer eye-piece was used with an optical microscope to determine the coating thickness of coated powder particles after mounting, grinding, polishing and etching with a two-percent nital solution.

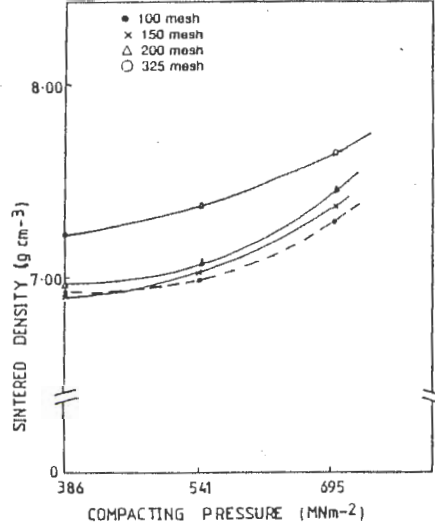


Figure 3. The effect of particle size distribution on sintered density at different compacting pressures.

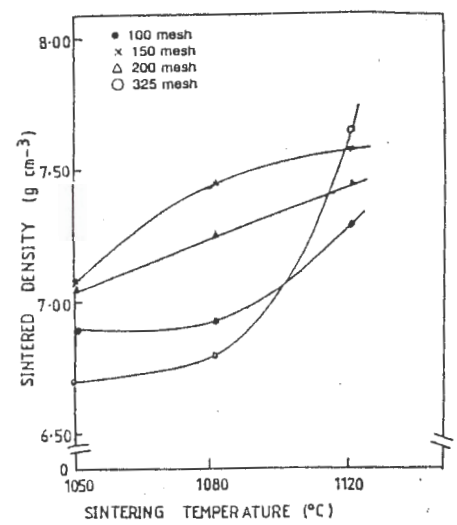


Figure 4. The effect of particle size distribution on sintered density at different sintering temperatures.

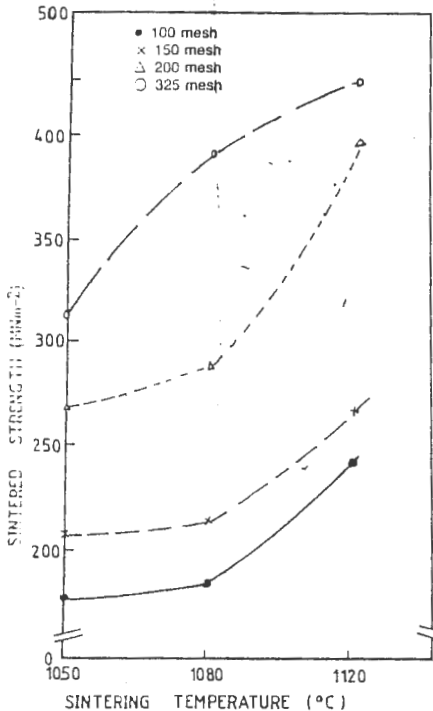


Figure 5. The effect of particle size distribution on sintered strength at different sintering temperatures.

volume diffusion or material transport. The greater surface area provided by the smaller particle size might create more paths for grain boundary/surface diffusion transport and hence more densification.

Figure 4 indicates that, in all the powder particle size ranges, sintered density increases with increasing temperature (Figure 5). However, the increase appears to have a random character, particularly at lower temperatures (1050 and 1080°C). The temperature needed to cause nickel diffusion into the iron matrix for homogenization and densification may not be attained. Additionally, because the powder surface area and apparent density differ, the extent of diffusion may vary. Inconsistent densification may also be due to the influence of nickel content, which is expected to be different in the different powder compacts and at different temperatures. Different individual particle sizes, as classified into mesh numbers, cause the surface areas and apparent density to vary. Depending on the operating temperature, the plated nickel might therefore be expected to diffuse at different rates into the iron matrix at different concentrations. The impurity effect associated with oxides and/or surface contamination can inhibit sintering and thereby reduce density.

#### HOMOGENEITY AND NICKEL CONTENT

The homogeneity of the sintered compacts results from proper alloying. The evolution of a homogeneous alloy structure for a completely solid-state sintering system is diffusion related. The degree of alloying increases greatly with a relatively small increase in sintering temperature. As evidenced by the micrographs and the mechanical and physical properties, the degree of homogeneity or alloying increases with smaller particle sizes.

The phosphorus content of the sintered compacts (0.38%) might contribute to stabilizing the  $\alpha$  phase by increasing the solubility of nickel. It might also contribute to lowering of the melting point and, therefore, cause an increase in the diffusion coefficient<sup>10,11</sup> resulting in improved homogeneity and correspondingly better properties. The increase in nickel content at smaller particle sizes seems to improve the densification of the sintered compacts. Similar results have been reported elsewhere.<sup>12,13</sup> The increase in densification may be explained by the slightly higher diffusivities of nickel in fine powder as compared to coarse powder particles because of the greater surface or interfacial area. It may thus be concluded that the intrinsic diffusivities of iron and nickel in Fe-Ni alloys increase with increasing nickel concentrations.<sup>14</sup>

#### ABOUT THE AUTHOR

C.A. Loto received his Ph.D. in metallurgical engineering from the University of Manchester Institute of Science and Technology, Manchester, U.K., in 1984. He is currently a lecturer in the Metallurgical and Materials Engineering Department at the University of Ife, Ile-Ife, in Nigeria.

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#### References

1. W. Canning, *The Canning Handbook on Electroplating*, 20th Edition (1978), pp. 425-429.
2. L.F. Spencer, *Metal Finishing*, Oct. and Nov. (1974).
3. R.M. Burns and W.W. Bradley, *Protective Coatings for Metals*, Reinhold (1947), pp. 229-230.
4. A. Brenner and G.E. Riddell, *Proc. Am. Electroplaters' Soc.* (1947), p. 156.
5. C.H. Minjer and A. Brenner, *Plating*, 44 (1975), p. 1297.
6. R.A.F. Hammond, "Pt. 1," *Metal Finishing J.*, Vol. 18, Nos. 6-9 (1972).
7. D.A. Fanner and R.A.F. Hammond, *Trans. IMF*, 36 (1959), p. 32.
8. H. Dixon, A.J.F. Fletcher and R.T. Cundill, *Powder Metallurgy*, 21, 3 (1978), p. 131.
9. P. Lindskog and G. Skoglund, 3rd Eur. P/M Symp. Conf. Suppl. Pt. 1, Nov. (1971), p. 375.
10. A.S. Doan, Jr., and J.I. Goldstein, *Met. Trans.*, 1 (1970), p. 1759.
11. T.R. Heyward and J.I. Goldstein, *Met. Trans.*, 4, 10 (1973), p. 2335.
12. F. Thummler and W. Thomma, *Met. Review*, 115, 12 (1967), p. 69.
13. W.V. Knapp, *Progress in P/M*, 16 (1960), p. 84.
14. Wanin and C.R. Kohn, *Int. J. of P/M & Powder Technology*, 15-16 (1979), p. 64.