# EFFECT OF NICKEL PARTICLE SIZE ON PROPERTIES OF P/M STEELS

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## ABSTRACT

The effect of Ni particle size on the properties of 4%Ni-0.5%C steels in the as-sintered condition has been examined. Ni powders were chosen from two manufacturing methods: Ni carbonyl and electrolytic Ni technologies. Nickel carbonyl powder technology is well suited for the production of fine, roughly spherical or filamentary Ni powders, typically less than 15 um in diameter; whereas electrolytic Ni powders are normally relatively coarse and dendritic in morphology. Finer Ni particle size and improved Ni distribution increase mechanical properties of P/M steels.

## INTRODUCTION

Powder metal steel parts derive their properties from a combination of materials and processing. Ni and Cu are the two most commonly used alloying elements for improving the properties of P/M steels. Both Ni & Cu strengthen and harden steel by solid solution strengthening. With Ni or Cu in the steel, the eutectoid carbon ( C ) level is lowered, therefore less C is needed for strengthening ferrite, avoiding embrittlement of steel at higher C content. In addition, hardenability is increased by lowering upper and lower critical transformation temperatures, permitting slower cooling to form hard, strong phases such as martensite. Cu has a slightly higher hardening effect than Ni due to the limited diffusion of Ni in the as-sintered condition. Ni has a greater effect on improving elongation and impact energy; a 2% Ni addition to a plain 0.5% carbon steel can increase toughness by four times. Standard data generated by the Metal Powder Industries Federation in North America shows that tensile strength and hardness of plain carbon steels can be increased by up to 80% with additions of either 2% Ni or 2% Cu additions [1].

In the heat-treated condition all steels have relatively low ductility and toughness. Cu has little strengthening effect and can in fact embrittle steel, in particular prealloyed Fe-Mo steels, while Ni greatly improves strength and toughness. Nickel gives the highest strength, with slightly lower hardness due to the persistence of soft Ni-rich phases in the steel microstructure. The role of these Ni-rich phases continues to be a controversial subject in the international P/M industry. Recent work by the authors on an extra-fine grade of Ni powder suggests that the distribution, size and resulting structure of Ni-rich phases can have a very large effect on both the magnitude and consistency of the physical properties of Ni P/M steels [2-4].

## EXPERIMENTAL

Three carbonyl Ni powders were used in this study: Inco® Type 123 PM, 255 and 110 PM herein referred to as Standard, Fine and Extra-fine grades. Performance of these carbonyl powders was compared to an electrolytic Ni powder produced by Jinchuan Group Limited in China. Standard and extra-fine grade Ni powders are discrete and differ primarily in particle size and surface morphology:

- *Standard Ni powder particle* is discrete with 8 um diameter and spiky surface.
- *Extra-fine Ni powder particle* is discrete with 1.5 um diameter and smooth surface.
- *Fine grade powder* is filamentary, with primary Ni particle size of approximately 2-3 um. Although not commonly used in P/M steels outside of China, the Chinese P/M market uses this Ni powder frequently due to its availability and the need for an export license to import discrete Ni powders.
- *Electrolytic Ni powder* is relatively coarse with a dendritic morphology. Mean particle diameter ($d_{50}$) is 37 um. Particle distribution is wide, with a $d_{90}$ of 72 micrometres as measured by laser diffraction.

Tensile and transverse rupture bars were pressed at 550 MPa compaction pressure, then sintered in a 10% hydrogen / 90% nitrogen atmosphere laboratory tube furnace at 1120 °C for 30 minutes.

## RESULTS & DISCUSSION

### Ni distribution in SEM microstructure

Ni is often added to P/M steels in quantities not exceeding 4% by weight, with 2% being most common. Scanning electron microscope images of Ni-rich phases in a 4%Ni-0.5%C steel are shown in Fig.1, comparing steels made with standard and electrolytic Ni powder. Ni-rich phases are light in colour, with porosity represented by irregular black areas and the medium grey steel microstructure consisting mainly of pearlite and ferrite. Note that Ni-rich phases tend to be associated with porosity, a consequence of the slow diffusion of Ni and the location of Ni powder on the original surface of Fe particles. The light Ni-rich regions are seen to be larger in the Electrolytic Ni steel; they are also more intense in colour, indicating higher Ni content.

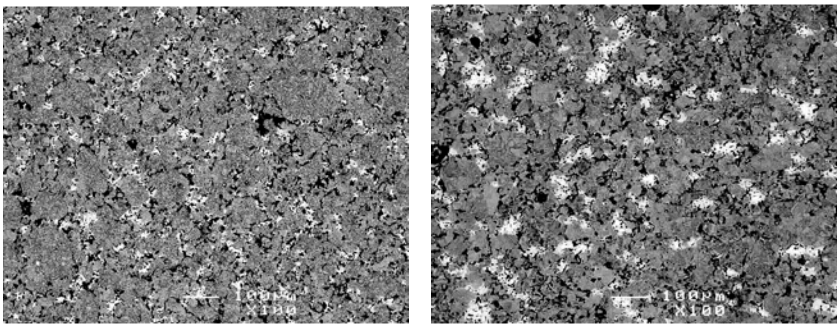


Fig. 1. Low-magnification SEM images of representative cross-sections of 4%Ni-0.5%C steels with Standard Ni powder (left) and Electrolytic Ni powder additions (right).

### Diffusion of Ni during sintering

Surface area of Ni-rich areas in 4%Ni-0.5% C steels was determined by applying a grid to SEM images taken at 200X magnification. Typically 7 to 14 Ni-rich areas per field and three fields per sample were measured. Surface areas of Ni-rich phases ranged from 100 to 470 square um, for steels made with extra-fine and electrolytic Ni powders respectively. Standard deviation increased with increasing surface area, such that the variability in surface area of Ni-rich regions was 5 times higher for electrolytic Ni powder than for all carbonyl Ni powders. Results are shown in Figure 2a.

As would be expected, the larger the Ni-rich region, the higher the Ni content. Average Ni content of Ni-rich phases in 4% Ni steels was measured by X-ray dispersive spectrometry in the SEM at 500X magnification. Over an average of three fields each 185 x 170 micrometers in surface area, the Ni content ranged from under 10 to over 30% for Ni-rich phases in steels made with extra-fine and electrolytic Ni powders respectively. Standard deviation of Ni concentration in the Ni-rich area varied from 2% for extra-fine Ni, to 12% for electrolytic Ni. As the Ni content of Ni-rich phases approaches values less than 5%, Ni-rich austenite is no longer stable. The structure of Ni-rich phases is therefore dependent on both Ni content and cooling rate. Evidence of Ni-rich martensite formation in as-sintered steels made with extra-fine Ni powder has been observed in other works by the authors [2-4]. Results are shown in Figure 2b.

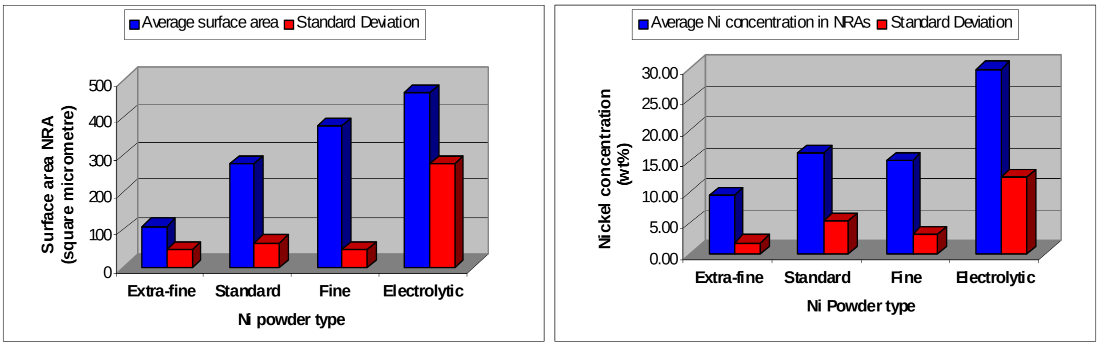



Figure 2. (2a, left side) Mean size of Ni-rich areas (NRA) in different 4%Ni-0.5%C steels. (2b, right side) Average Ni concentration & standard deviation of Ni-rich areas (NRA) in different 4%Ni-0.5%C steels.

By combining the surface area and Ni content measurements, a semi-quantitative method has been developed to estimate diffusion rates of the various Ni powders in P/M steels. Figure 3 plots the amount of Ni diffused in the steel (%) vs. the sintering time. The samples were sintered at 1120 °C for 30 and 60 minutes. After conventional sintering for 30 minutes, over 80% of extra-fine Ni has diffused. whereas only 50 to 60 % of fine and standard Ni powders has diffused. Fine Ni powder tends to have slightly less average diffusion than standard Ni powder using this calculation method, as a result of the greater tendency of fine filamentary Ni powder to agglomerate. Only 25% of electrolytic Ni powder has diffused at the same sintering conditions.

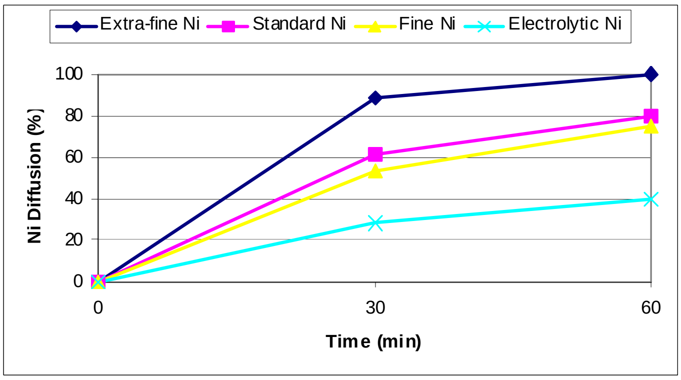



Figure 3. Ni diffusion vs. sintering time for 4%Ni-0.5%C steels made with different Ni powder additions.

<u>Powder properties of P/M steel blends & Green properties of P/M steels</u>

Laboratory scale 500 g batches of plain Fe powder (Hoeganaes Ancorsteel 1000B) were mixed with 4 wt% Ni powder, 0.6wt% C and 0.75% lubricant (Lonza Acrawax C) in a Turbula mixer for 20 minutes. Hall flow was measured for fresh powder batches, with no discernable difference noted between Ni powder types. Apparent density as determined by Scott Volumeter was slightly higher for finer particle size. Green density was highest for the electrolytic powder, as would be expected for the largest particle size. Green transverse rupture strength was also highest for the steel containing the coarsest Ni powder. However the next highest value for both green density and strength was for the extra-fine Ni powder. The reason for this behaviour is thought to be related to the way extra-fine Ni powder adheres to the irregular surface of Fe particle, increasing green density and strength relative to standard or fine grade Ni powders. Results are shown in Table1a.

<u>Mechanical properties of P/M steels</u>

As-sintered Ultimate Tensile strength (UTS) and Transverse Rupture strength (TRS) increased with decreasing Ni particle size. Standard deviation of UTS was highest for standard Ni powder at 66 MPa and averaged approximately 20 MPa for the other three Ni powders. Standard deviation of TRS ranged from 3

MPa in extra-fine Ni steels to 53 MPa in electrolytic Ni powder steels. Table 1b summarizes the sintered properties of steels made with the four Ni powders in this study. With decreasing Ni particle size:

- As-sintered TRS increased from 710 to 990 MPa
- UTS increased from 390 to 520 MPa
- Average elongation of 2.7% was roughly equivalent for all steels.

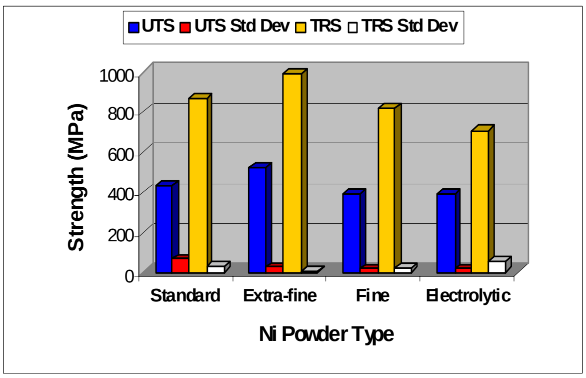
Apparent hardness increased by 30% from 65 to 84 HRB for electrolytic and extra-fine Ni steel respectively.

Table 1. (1a, left) Powder properties of 4%Ni-0.5%C steel blends; (1b, right) mechanical properties of as-sintered 4%Ni-0%5 steels.

| *Ni powder Type* | Sinter Density ($g/cm^3$) | TRS (MPa) | UTS (MPa) | Elong (%) | App. Hardness (HRB) |
|---|---|---|---|---|---|
| Extra-fine | 7.10 | 990 | 520 | 2.6 | 84 |
| Standard | 7.05 | 870 | 430 | 2.6 | 79 |
| Fine | 7.04 | 820 | 390 | 2.8 | 71 |
| Electrolytic | 7.09 | 710 | 390 | 2.7 | 65 |

| *Property* | Standard Powder | Extra-fine Powder | Fine Powder | Electrolytic Powder |
|---|---|---|---|---|
| Hall Flow (s/50g) | 34 | 34 | 34 | 34 |
| Apparent Density ($g/cm^3$) | 3.07 | 3.1 | 3.05 | 3.01 |
| Green Density ($g/cm^3$) | 6.98 | 7.02 | 6.95 | 7.09 |
| Green Strength (MPa) | 10.9 | 11.2 | 10.5 | 12 |

UTS and TRS are summarized in Fig. 4. The increased diffusion of Ni powder as particle size decreases can be measured in microindentation hardness profiles of sintered Ni steels. In Figure 5, microindentation hardness was measured along the sample length for as-sintered 4%Ni-0.5%C steels. Hardness was measured using the Vicker's scale with 500 g applied load. Average microindentation hardness of steels made with carbonyl Ni powders was 15 to 20% higher than electrolytic Ni steel. Standard deviation of microindentation hardness was approximately 4 X higher for electrolytic Ni steels.



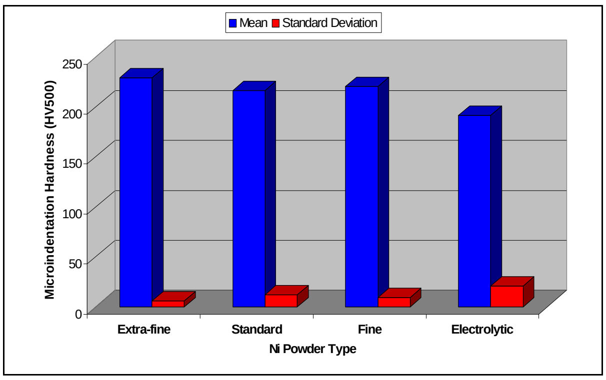
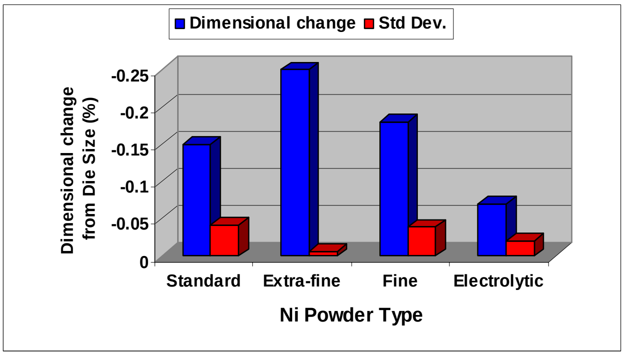




Fig.4 (left side) UTS and TRS data of 4%Ni-0.5%C steels with different Ni powders;
Fig.5 (right side) Microindentation hardness values of 4%Ni-0.5%C steels with different Ni powders.

Dimensional change properties

The fact that Ni steels shrink during sintering, leading to densification of P/M steel parts is well known. Ni is often added to Cu-steels to control swelling. The shrinkage of Ni steels is controlled by the extent of Ni diffusion into Fe during sintering. Additional shrinkage can be obtained by higher sintering temperatures or longer sintering time and also by using finer Ni powder. In Figure 6 not only the highest shrinkage, but also the lowest standard deviation in per cent change from die size is obtained with extra-fine Ni powder. Provided good mixing practices are employed to avoid agglomeration, fine Ni powder can sometimes used by the P/M parts manufacturer in place of standard Ni powder to control dimensional change.



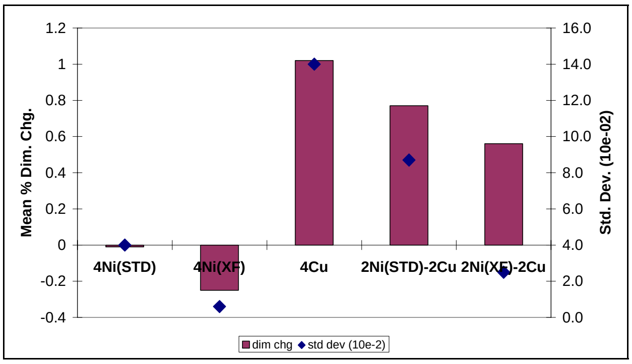




Fig. 6 (left) Per cent change and standard deviation of change from die size 4% Ni-0.5% C steels.
Fig. 7 (right) Mean % Dimensional change & standard deviation of 2%Ni-2%Cu-0.5%C steels. (STD = Standard Ni, XF = Extra-fine Ni).

Ni-Cu steels

An added benefit of improved dispersion of extra-fine Ni powder in the steel microstructure is the influence Ni has on the distribution of other alloying elements such as standard grade (-165 mesh) Cu powder. Cu has been found to preferentially associate with Ni during sintering [3]. Extra-fine Ni steels were found to have a much more uniform distribution of Ni and Cu than standard Ni steels. Apparent hardness was similar, however TRS was 920 vs. 830 MPa for the extra-fine vs. standard Ni powder steel respectively [4].

The improvement in distribution of Ni and Cu also has significant implications for dimensional control and part-to-part consistency. In Figure 7, dimensional change from die size measurements are shown for 4 Ni, 4 Cu and 2Ni-2Cu 0.5C steels made with extra-fine and standard Ni powder. Extra-fine Ni powder not only contributed to reduced swelling of the Cu-Ni steel after sintering, but also reduced the standard deviation between results by 75%.

## CONCLUSION

The benefits of using finer Ni powders in P/M steels include:

- Improved Ni distribution – increases uniformity of part composition and density distribution
- Increased Ni diffusion – increases hardenability and strength of steel
- Reduced variability in physical properties – improves consistency in part performance
- Improved dimensional precision – important for difficult to hold tolerances in larger parts.

The effectiveness of finer Ni carbonyl powders in improving mechanical properties of P/M steels more than compensates for the additional cost compared to coarser electrolytic Ni powder.

## REFERENCES


1. MPIF Standard 35 January 2005 Release, Materials Standards for P/M Structural Parts, 2003 Edition, p 33
2. S. Campbell, T. Singh, T.F. Stephenson, “Improved Hardenability of P/M Steels using Extra-fine Nickel Powder”, *Advances in Powder Metallurgy and Particulate Materials – 2004*, Part 7, pp. 105-115, Metal Powder Industries Federation, Princeton, NJ 2004
3. T. Singh, T.F. Stephenson, S. Campbell, "Nickel-Copper Interactions in P/M Steels", *Advances in Powder Metallurgy and Particulate Materials – 2004*, Part 7, pp. 93-104, Metal Powder Industries Federation, Princeton, NJ 2004
4. Stephenson, T.F., Singh T., Campbell, S.T., "Performance Benefits in Sintered Steels with XF-Ni powder", Proceedings of the World PM2004 Congress, Vienna, Austria, October 2004, Vol. 3 Sintered Steels, EPMA (European Powder Metallurgical Association)