Chemical Vapor Synthesis of Nonagglomerated Nickel Nanoparticles by In-Flight Coating

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ABSTRACT: Nickel (Ni) nanoparticles (NPs) prepared through vapor-phase synthesis (VPS) are preferred for multilayer ceramic capacitor electrodes due to their high purity and crystallinity advantages. Agglomerated Ni NPs are usually generated using VPS but are undesirable because they cause various problems such as low packing density and electrical shorts. This study proposes the use of coating-assisted chemical vapor synthesis (CVS) for agglomerate inhibition using NaCl or KCl as a coating agent. We have found that the agglomeration ratio, 34.40%, for conventional CVS, can be reduced to 4.80% in the proposed method by in-flight coating with KCl at 900 °C by image analysis using field-emission scanning electron microscopy. Furthermore, the X-ray diffraction and X-ray fluorescence analyses confirm that the NaCl and KCl coating agent can be removed by washing with distilled water. We believe that this coating process can be used to inhibit the formation of agglomerates during the CVS of Ni NPs.

INTRODUCTION

Nickel (Ni) nanoparticles (NPs) have been widely used in various applications, including electrodes, catalysts, batteries, and supercapacitors. More specifically, they have been used extensively as electrode materials for multilayer ceramic capacitors (MLCCs). Various methods, including wet chemical synthesis and vapor-phase synthesis (VPS), have been used to produce Ni NPs. Those prepared by VPS, including spray pyrolysis, physical vapor synthesis, and chemical vapor synthesis (CVS), are preferred for MLCC electrodes due to their high purity and crystallinity advantages. The formation of agglomerates during VPS is typical due to the high process temperature and absence of surfactants. Agglomerated particles are preferred in applications, such as catalysts and sensors, because they have a large surface area. However, such particles are undesirable for use in MLCC electrodes, as they cause various problems such as low packing density and electric shorts. Therefore, a classification process is performed to remove agglomerates from Ni NPs prepared by VPS, resulting in a low yield.

In VPS, particles are formed by nucleation, surface growth, and coagulation. The coagulation frequency increases when the temperature or number density of the particles increases or the mass of individual particles decreases. Coagulated particles usually evolve into spherical particles through full coalescence or into chain-like agglomerated particles through partial coalescence. Complete coalescence occurs with enough temperature and residence time and when the size of the particles is smaller than the critical size depending on the material. The trend toward increasing the capacitance and miniaturization of MLCCs has been driving a reduction in the Ni NP size requirements. If the particle size decreases while maintaining the production rate, the coagulation rate increases because the number density of particles increases exponentially. Thus, this miniaturization trend has caused the agglomerate formation to become more problematic.

Many researchers have studied and developed methods to inhibit agglomeration during VPS. Gas quenching has been studied most actively. However, the cost of this method is high due to excessive gas usage, and it is technically challenging to reduce the temperature rapidly and uniformly when the reactor scale is increased. Another promising method is vapor phase coating. In this method, Na and NaCl are used as a reducing agent and a coating, respectively, to produce NaCl-
coated NPs during flame synthesis. However, it is difficult to control this process because it is necessary to use highly reactive Na, and coating is performed by NaCl as a byproduct. We, therefore, chose to study the coating process using CVS, as shown in Figure 1, which should allow easier control by using a nonreactive coating agent such as NaCl and KCl.

**Theoretical Basis.** In this study, we tried to inhibit agglomeration using in-flight coating in the cooling section. The intent was to perform in-flight coating in a temperature range of $800 - 900^\circ C$. We considered metal chlorides as coating agent candidates for the following reasons. First, they have an appropriate vapor pressure for condensation to occur between 800 and 900 °C. Second, their thermal decomposition does not occur at a reactor temperature of 1000 °C. Third, we can easily remove them by water washing.

Among the various metal chlorides, we selected MgCl$_2$, LiCl, NaCl, KCl, ZnCl$_2$, and CaCl$_2$ as candidates from the following steps. First, metal chlorides reacting with H$_2$ and Ni were excluded, which eliminated chlorides such as FeCl$_3$, CuCl, and CoCl$_2$. Second, chlorides of radioelements were excluded. Third, chlorides of rare-earth elements were excluded due to their high price. Fourth, metal chlorides with a boiling point lower than 800 °C were excluded, resulting in the elimination of chlorides such as AlCl$_3$ and GaCl$_3$.

Gibbs free energy changes for the hydrogen reduction reactions of NiCl$_2(g)$, MgCl$_2(g)$, LiCl$_2(g)$, NaCl$_2(g)$, KCl$_2(g)$, ZnCl$_2(g)$, and CaCl$_2(g)$ were calculated by Thermo-Calc DB to determine reactivities of the selected candidates, as shown in Figure 2. Note that if a particular metal chloride does not react with H$_2$, it also does not react with Ni. Figure 2 shows that only NiCl$_2(g)$ should be expected to react with H$_2$.

![Figure 1. Schematic of coating-assisted CVS.](image1)

![Figure 2. Hydrogen reduction Gibbs free energies for various metal chlorides as a function of temperature.](image2)
reactor. Therefore, it is appropriate to use the smallest amount of the coating agent that can inhibit agglomeration. Among the metal chlorides, NaCl and KCl, which are earth abundant, were selected in this study to investigate the effect of in-flight coating on the formation of agglomerates during CVS. We determined that the agglomeration inhibition behavior according to the vapor pressure could be observed because the vapor pressures of NaCl and KCl differ more than twice at 900 °C.

RESULTS AND DISCUSSION

CVS of Uncoated Ni NPs. The field-emission scanning electron microscopy (FESEM) image of uncoated Ni NPs (sample 1) prepared by conventional CVS is shown in Figure 4a and the corresponding particle size distribution histogram is shown in Figure 4b. The count median diameter (CMD) and geometric standard deviation (GSD) of the Ni NPs in this sample were 75.60 nm and 1.24, respectively. The number of agglomerates consisting of 2, 3, 4, and 5 primary particles was identified as 38, 19, 6, and 3, respectively, and the agglomeration ratio was 34.40%. The CMD, GSD, and agglomeration ratios of samples 1–7 are summarized in Table 2.

Table 1. Samples Prepared in This Study

| sample number | feed rate of feedstock (g/h) | NaCl (wt %) | KCl (wt %) | vapor pressure of feedstock (kPa) | coating temperature (°C) |
|---------------|-----------------------------|-------------|------------|---------------------|-------------------------|
| 1             | 10                          |             |            | 0.04                | 800                     |
| 2             | 10.30                       | 2.91        | 0.04      | 800                 |
| 3             | 10.75                       | 6.99        | 0.11      | 850                 |
| 4             | 11.78                       | 15.06       | 0.24      | 900                 |
| 5             | 10.85                       | 7.83        | 0.09      | 800                 |
| 6             | 12.17                       | 17.83       | 0.23      | 850                 |
| 7             | 15.51                       | 35.49       | 0.54      | 900                 |

Table 2. CMD, GSD, and Agglomeration Ratio of Samples

| sample number | CMD (nm) | GSD | agglomeration ratio (%) |
|---------------|----------|-----|-------------------------|
| 1             | 75.60    | 1.24| 34.40                   |
| 2             | 81.80    | 1.25| 23.60                   |
| 3             | 64.50    | 1.24| 20.20                   |
| 4             | 63.70    | 1.33| 17.40                   |
| 5             | 74.20    | 1.25| 16.40                   |
| 6             | 72.10    | 1.28| 16.00                   |
| 7             | 71.90    | 1.33| 4.80                    |

The X-ray diffraction (XRD) result of sample 1 in Figure 4c shows peaks of Ni matching with JCPDS 04-0850. The X-ray fluorescence (XRF) result of sample 1 in Figure 4d shows that the Ni and Cl contents were 98.47 and 1.53 wt %, respectively. The XRF results of samples 1–7 are summarized in Table 3. It is assumed that NiCl₂ was deposited on the surface of the Ni

Figure 3. Saturated vapor pressure of various metal chlorides as a function of temperature.

Figure 4. (a) FESEM image, (b) particle size distribution histogram, (c) XRD, and (d) XRF results of uncoated Ni NPs (sample 1) prepared by conventional CVS.
NPs as an unreacted residual precursor. As this is a trace amount, it was likely not detected by XRD.

Table 3. XRF Results of Samples

| sample number | Ni (wt %) | Na (wt %) | K (wt %) | Cl (wt %) | Ni (wt %) | Na (wt %) | K (wt %) | Cl (wt %) |
|---------------|-----------|-----------|----------|-----------|-----------|-----------|----------|-----------|
| 1             | 98.47     | 1.53      |          | 0.36      | 99.64     | 0.36      |          |           |
| 2             | 95.20     | 0.97      | 3.83     |           |           |           |          |           |
| 3             | 91.10     | 2.34      | 6.57     |           |           |           |          |           |
| 4             | 86.50     | 4.30      | 9.13     | 0.61      | 99.30     | 0.61      |          |           |
| 5             | 91.61     | 3.97      | 4.43     |           |           |           |          |           |
| 6             | 81.17     | 8.93      | 9.90     |           |           |           |          |           |
| 7             | 64.69     | 17.96     | 17.36    | 0.11      | 99.89     | 0.11      |          |           |

CVS of NaCl-Coated Ni NPs. Figure 5a–c shows SEM images of NaCl-coated Ni NPs (samples 2–4) prepared by coating-assisted CVS. The size distribution histograms of the Ni cores are shown in Figure 5d–f. When the coating temperature was 800 °C, the CMD and GSD values for the coated NP Ni cores were 81.80 nm and 1.25, respectively. The number of agglomerates consisting of 2, 3, 4, and 5 primary particles was identified as 34, 15, 5, and 0, respectively, and the agglomeration ratio was 23.60%. When the coating temperature was 850 °C, the CMD and GSD values were 64.50 nm and 1.24, respectively. The number of agglomerates consisting of 2, 3, 4, and 5 primary particles was identified as 27, 13, 2, and 0, respectively, and the agglomeration ratio was 20.20%. When the coating temperature was 900 °C, the CMD and GSD values were 63.70 nm and 1.33, respectively. The number of agglomerates consisting of 2, 3, 4, and 5 primary particles was identified as 27, 9, 1, and 0, respectively, and the agglomeration ratio was 17.40%.

The agglomeration ratio decreased from 23.60 to 17.40% as the coating temperature was increased from 800 to 900 °C. It was determined that the agglomeration ratio, which was 34.40% when not coated, was reduced to 17.40% by coating with NaCl. However, the agglomeration ratio is still high even when Ni NPs are coated with NaCl. To further reduce the agglomeration ratio, it may be possible to increase the coating temperature or use a metal chloride with a vapor pressure higher than that of NaCl.

The XRD results of samples 2–4 in Figure 6a show peaks of Ni and NaCl matching with JCPDS 04-0850 and 72-1668.
respectively. The XRF results are shown in Figure 6b. When the coating temperatures were 800, 850, and 900 °C, the Ni contents were 95.20, 91.10, and 86.56 wt %, the Na contents were 0.97, 2.34, and 4.30 wt %, and the Cl contents were 3.83, 6.57, and 9.13 wt %, respectively. These results show that as the temperature increased, the amount of the remaining coating agent also increased.

**CVS of KCl-Coated Ni NPs.** FESEM images of KCl-coated Ni NPs (samples 5–7) prepared through coating-assisted CVS are shown in Figure 7a–c. The size distribution histograms of the Ni cores are shown in Figure 7d–f. When the coating temperature was 800 °C, the CMD and GSD values for the coated NP Ni cores were 74.20 nm and 1.25, respectively. The number of agglomerates consisting of 2, 3, 4, and 5 primary particles was identified as 27, 8, 1, and 0, respectively, and the agglomeration ratio was 16.40%. When the coating temperature was 850 °C, the CMD and GSD values were 72.10 nm and 1.28, respectively. The number of agglomerates consisting of 2, 3, 4, and 5 primary particles was identified as 28, 8, 0, and 0, respectively, and the agglomeration ratio was 16.00%. When
the coating temperature of KCl was 900 °C, the CMD and GSD values were 71.9 nm and 1.33, respectively. The number of agglomerates consisting of 2, 3, 4, and 5 primary particles was identified as 9, 2, 0, and 0, respectively, and the agglomeration ratio was 4.80%.

The agglomeration ratio decreased from 16.40 to 4.80% as the coating temperature was increased from 800 to 900 °C. It was determined that agglomeration could be effectively inhibited by coating with KCl. As the saturated vapor pressure of KCl is higher than that of NaCl, the distance between the nickel cores is sufficient to inhibit agglomeration when a relatively large amount of KCl is deposited.

Figure 8 shows FESEM and energy-dispersive X-ray spectroscopy (EDS) results of sample 7. It was observed that the KCl coating layer surrounding the Ni core looks transparent. This can be explained because the penetration depth is large when the accelerating voltage is high and the atomic number is low.30,31

The XRD results of samples 5–7 in Figure 9a show the peaks of Ni and KCl matching with JCPDS 04-0850 and 41-1476, respectively. The XRF results are shown in Figure 9b. When the coating temperatures were 800, 850, and 900 °C, the Ni contents were 91.61, 81.17, and 64.69 wt %, the K contents were 3.97, 8.93, and 17.96 wt %, and the CI contents were 4.43, 9.90, and 17.36 wt %, respectively. Again, more of the coating agent remained as the temperature increased, but the effect was more pronounced with KCl.

**CONCLUSIONS**

We carried out the coating-assisted CVS using NaCl and KCl as agglomeration inhibitors. Compared to conventional CVS with an agglomeration ratio of 34.4%, NaCl and KCl inhibitors reduced the ratio to 17.4 and 4.8%, respectively, when processed at 900 °C. KCl is a more effective agglomeration inhibitor than NaCl, presumably due to its higher vapor pressure. Therefore, we expect that a further increase in the inhibitor vapor pressure would be helpful during NP manufacturing with a higher number density. The NaCl and KCl inhibitors used in this study were easily removed with distilled water. Thus, we believe that coating-assisted CVS can inhibit the formation of agglomerates in the manufacture of Ni NPs.

**EXPERIMENTAL SECTION**

**Preparation of Feedstock for the Synthesis of Uncoated Ni NPs.** NiCl2·6(H2O) (Alfa Aesar, 98%) was dissolved in deionized water, and the resulting solution was spray-dried at 180 °C (B-290, BUCHI Labortechnik). NiCl2·2(H2O) powder collected from a spray-dryer was dried at 250 °C under a pressure of 10⁻⁵ Torr to obtain an anhydrous NiCl2 powder.

**Preparation of Feedstock for the Synthesis of Coated Ni NPs.** NiCl2·6(H2O) and NaCl (Sigma-Aldrich, 99.5%) or KCl (Sigma-Aldrich, 99.0%) were dissolved together in distilled water followed by spray-drying of the mixed solution at 180 °C. The composite powder collected from the spray-drier was dried at 250 °C under a pressure of 10⁻⁵ Torr.

**CVS of Uncoated and Coated Ni NPs.** The vertical hot-wall reactor shown in Figure 1 was used for CVS at a fixed heating zone temperature of 1000 °C (Figure 1a). Because the coating...
vapor pressure of NiCl₂ and the H₂ reduction rate are proportional to the temperature, increasing the process temperature has the advantage of increasing the Ni powder production rate. However, due to the limit of the operating temperature of the reactor material, quartz, all process temperatures in this study were performed at 1000 °C with a precision of ±2 °C. The feedstock was introduced to a reactor by a powder feeder (Fine Techniques) with an N₂ carrier gas flow rate of 5.6 standard liter per minute (SLM). Table 1 shows that the coating agent content and feed rate changed with the coating temperature. The amount of the coating agent content was determined by the saturated vapor pressure at the coating temperature. Note that the feed rate was increased to increase only the injection amount of the coating agent while maintaining the injection amount of NiCl₂ at 10 g/h. H₂ reducing gas was injected directly into the particle formation section at a flow rate of 0.5 SLM (Figure 1b). The temperature decreased through the cooling section due to air cooling of the reactor outer wall (Figure 1c).

Metal chlorides evaporate in the top section while H₂ reduction of NiCl₂ vapor occurs in the particle formation section, leading to nucleation, growth, and coagulation of Ni NPs. Complete coalescence of coagulated NPs mainly occurs in the particle formation section, and partial coalescence primarily occurs in the cooling section. The in-flight coating was performed in this cooling section to inhibit partial coalescence in the coating temperature range of 800−900 °C.

Ni NPs produced by CVS were retrieved from the collector and characterized by FESEM (Quanta 250 FEG, FEI), XRD (Empyrean, Panalytical), and XRF (M4 TORNADO, Bruker). Each sample was sputter-coated with a thin film of Au−Pd before FESEM analyses to improve the resolution on the FESEM images. The inner electrode of MLCC is made of Ni NPs and its thickness is only hundreds nm. Hence, the average particle size and size distribution of the Ni NPs are important and were expressed as CMD and GSD, respectively. CMD and GSD were determined by a randomly sampling of 200 particles from FESEM images. The agglomeration ratio was calculated by dividing the number of primary particles constituting agglomerates by the sum of the primary particles constituting agglomerates plus non-agglomerated particles, as determined by the randomly sampling of more than 500 particles from FESEM images. The content ratio and phase of the coating agent and Ni were determined by XRD analysis. The residual amount before and after the removal of the coating agent was determined by XRF analysis.

**Removal of Chlorides.** Samples 1, 4, and 7 were sonicated in distilled water (Q500, Qsonica) for the removal of chlorides. Three samples were selected to determine whether the coating agent could be removed. Sample 1 without using a coating agent and samples 4 and 7 with the highest amount of each coating agent were selected as a control. After washing, NPs were obtained by centrifugal separation at 10,000 rpm and characterized by XRD and XRF. CMD and GSD were determined by randomly sampling of 200 particles from the FESEM image of washed sample 7. Also, the agglomeration ratio was determined by randomly sampling more than 500 particles from FESEM images.

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Notes

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