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# A study on synthesis of fine size cobalt powder from fluoride compounds

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### Abstract

Research studies were carried out to prepare extra fine and high purity cobalt powder from synthetic cobalt chloride solutions without the use of hydrogen. The two major steps involved in the process were, first, precipitation of an appropriate ammonium cobalt fluoride salt and second, its thermal decomposition to cobalt powder. The optimum conditions for precipitation of  $NH_4CoF_3$  were found to be, temperature of 30 °C, 100% excess  $NH_4F$  and a solution pH of 5. Cobalt metal powder was prepared from  $NH_4CoF_3$  in a closed reactor and decomposition cum reduction temperature and time were optimized to 550 °C and 6 h, respectively. Detailed XRD studies identified all the intermediate and final products. The powder was further characterised by Chemical and SEM analysis. SEM analysis revealed the formation of 100–500 nm cobalt powder having spherical/round morphology.

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# 1. Introduction

Conventional production methods for cobalt and nickel powders commonly use a multistage process involving hydrogen reduction of aqueous salt solution of oxide, chloride or sulphate under pressure and high temperature [1,2]. This reduction process yields a powder with a purity of 99.6% and a particle size of around 5 µm. Studies are also reported on direct hydrogen reduction of powders of oxides, chlorides and sulphates [3]. Of these, oxide intermediates only resulted in cobalt powder, of size in the range of  $2-5 \,\mu\text{m}$ . Thermal decomposition of cobalt oxalate under reducing atmosphere of hydrogen [4] produces a cobalt powder of 99.8-99.9% purity with a crystal size of around  $1-2\,\mu m$ , but requires preconditioning due to highly pyrophoric nature and also suffers from a poor shelf life. Metal powders of particle size below 1 µm are mostly produced by many physical and chemical methods. Among the chemical methods, the reduction of a metal salt or oxide by an appropriate reducing agent is frequently employed. The size, shape and purity of the product largely depend on the type, purity of metallic precursor, reducing agent and conditions of, precipitation and reduction. However, Recently, Dong Jin Kin [5] reports on the preparation

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of 400 nm cobalt powder from Co(OH)<sub>2</sub> solution, by hydrogen reduction in presence of palladium catalyst. Considerable work has also been carried out on the preparation of fine size cobalt powder by several chemical methods such as carbonyl pyrolysis [6] and solution phase metal salt reduction [7–9]. For example, Zhu et al. [10] reports aqueous reduction of cobaltous hydroxide slurry under hydrogen reduction conditions using 2 hydroxy-4- (1-methylheptyl) benzophenone oxime to prepare nano-crystals of cobalt. Most of the reported work on submicron cobalt powder is from very lean solutions using complex organic compounds.

Cobalt metal and its compounds have found a wide range of applications. However, cobalt powder of particular size, purity and morphology find its application mainly in cemented carbide tools. Metallic materials produced from nano-size high purity powders offer new physical and mechanical properties and applications. In India, cobalt powder is needed for its fabrication to pellets  $(1 \text{ mm} \times 1 \text{ mm})$  and slugs  $(6 \text{ mm} \times 25 \text{ mm})$ , of 96% theoretical density for its ultimate conversion to gamma radiation source for cancer treatment. Most of the requirements of cobalt powder in India is met by imports. India imported about 36 tonnes of cobalt in the form of powders and flakes in the year 2002–2003 [11]. Although cobalt metal powder has proven applications in cemented carbide tools, meaningful data and technical details are not available in open literature to prepare cobalt metal powder for the above-cited distinguished applications.

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Fig. 1. Experimental set-up for decomposition of ACF to cobalt powder.

This paper describes an alternate method of cobalt powder production from an ammonium fluoride salt of cobalt, using chemically bound hydrogen as reductant. In comparison to most of the conventional powder production processes, this environmentally acceptable process, offers additional improvements including improved purity, reduced grain size, non-oxidized and non-inflammable powders.

In the absence of any valuable and meaningful data sighted in the literature, on preparation of ammonium cobalt fluoride, decomposition to cobalt fluoride and ultimately to fine metal powder, the paper attempts to provide an elaborate insight into the same. Characterization studies of powder by XRD, SEM, chemical analysis have also been incorporated.

#### 2. Experimental description

In this research, cobalt powder of different sizes is prepared from synthetic cobalt chloride solutions, by varying different parameters, of precipitation and decomposition of the cobalt salt. Particle size and purity were taken to be the most important characteristics of the product in conjunction with the yield of the reactions involved. In a typical procedure for precipitation of ammonium cobalt fluoride, 100 ml CoCl<sub>2</sub> (1 M) was adjusted to the required pH and precipitated with ammonium fluoride dissolved in minimum amount of water. The solution was maintained under constant stirring for 30 min at the required temperature. The light pink amorphous precipitate was filtered without washing, dried at a temperature less than 100  $^{\circ}$ C, weighed and then used as feed material for preparation of cobalt powder in the next stage.

The decomposition of the salt to Co powder was carried out in a resistance heating furnace with horizontal retort, as shown in Fig. 1. The air dried ammonium cobalt fluoride in batches of 10 g was taken in a closed molybdenum reactor and placed inside the retort. Firstly, argon was passed for half an hour in order to drive away any air inside the retort. Then the furnace was closed from both sides and maintaining slight argon pressure inside, heated to an inside temperature of 550 °C. After heating for the required time, the side valve was opened to release the excess fluoride to an ammonia collector. The retort was then cooled thoroughly and the product removed and weighed. The products were subsequently characterised by X-ray diffraction, scanning electron microscopy and chemical analysis.

# 3. Results and discussion

Chemical precipitation/reduction are a frequently used method for the preparation of metal powders of submicron sizes. However, the conditions for precipitation and subsequent decomposition of complex ammonium cobalt fluoride is not adequately described in literature [12]. The chemical reactions involved in the formation of synthetic ammonium cobalt fluoride from cobalt chloride by ammonium fluoride is based on the following reaction [2]:

$$CoCl_2 + 3NH_4F \rightarrow NH_4COF_3 + 2NH_4Cl$$
 (1)

It is assumed that the following sequence of chemical reactions take place during the course of preparation of cobalt metal powder [12,13]:

$$3NH_4CoF_3 \rightarrow 3CoF_2 + 3NH_3 + 3HF$$
(2)

$$2\mathrm{NH}_3 \to \mathrm{N}_2 + 3\mathrm{H}_2 \tag{3}$$

$$NH_3 + HF \rightarrow NH_4F$$
 (4)

$$3\text{CoF}_2 + 3\text{H}_2 \rightarrow 3\text{Co} + 6\text{HF}$$
 (5)

Put simply it is a one-stage manufacture of metal powder from an intermediate cobalt fluoride salt. The decomposition had to be carried out in a closed reactor maintained under pressure of the order of  $1.5 \times 10^5$  Pa, so as to prevent any escape of ammonia from the system, generated during heating of NH<sub>4</sub>CoF<sub>3</sub>. Thermal decomposition of CoF<sub>2</sub> under the operating conditions of the reactor is not favorable. However, H<sub>2</sub> reduction of CoF<sub>2</sub> is thermodynamically feasible and produces cobalt metal powder. Hydrogen required for the reduction is generated in situ in the system by the decomposition of ammonia. It is only the chemical reduction reaction shown in reaction (5) results in generation of cobalt metal powder. The reaction products are recycled except for nitrogen, which is vent out. The overall reaction can thus be expressed as

$$3NH_4CoF_3 \rightarrow 3Co + NH_4F + 8HF + N_2 \tag{6}$$

The decomposition of ammonium cobalt fluoride to intermediate products (CoF<sub>2</sub>) as per reaction (2) has only been supported by TG-DTA curves given in Fig. 2. The appearance of endotherm at 160 °C indicate loss of water of crystallization of the salt, followed by decomposition to cobalt fluoride at 320 °C. The appearance of two small peaks at around 245 and 260 °C might be on account of formation of successive lower ammonium cobalt fluorides. This is because the reaction takes place in several stages before the formation of CoF<sub>2</sub>. The intermediate CoF<sub>2</sub> phase, is further confirmed by XRD (Fig. 3). On further heating to 550 °C, chemically bound hydrogen from ammonia reduces the cobalt fluoride to metallic cobalt.



Fig. 2. TG-DTA graph for decomposition of ammonium cobalt fluoride to cobalt fluoride.

The two major stages as indicated above involved in the preparation of cobalt metal powder from cobalt chloride solutions are:

- i. synthesis of anhydrous ammonium cobalt fluorides;
- ii. thermal decomposition of ammonium cobalt fluoride.

The findings of the entire study are therefore discussed accordingly.

#### 3.1. Synthesis of ammonium cobalt fluoride

Due to lack of published literature and relevant technical data on preparation of ammonium cobalt fluoride from cobalt chloride, a thorough study was initiated to identify the optimum conditions. The preliminary studies indicated the presence of different fluoride salts of cobalt during precipitation. However, the studies had been focused on precipitation of one single



Fig. 3. X-ray diffraction analysis of the intermediate cobalt fluoride powder produced at 320 °C.

| Table 1                     |  |
|-----------------------------|--|
| Effect of ammonium fluoride |  |

| S. no. | $NH_4F(g)$  | ACF (g) | Co in filtrate (g) (unreacted) |
|--------|-------------|---------|--------------------------------|
| 1      | Stoic       | 7.20    | 4.2                            |
| 2      | 50% excess  | 8.50    | 3.8                            |
| 3      | 70% excess  | 10.00   | 3.4                            |
| 4      | 90% excess  | 12.00   | 3.0                            |
| 5      | 100% excess | 14.50   | 2.38                           |
| 6      | 110% excess | 14.20   | 2.4                            |

Co = 1 M (100 ml), pH 5, temperature =  $60 \circ C$ .

cobalt ammonium fluoride. This is because of the differences in thermodynamics and kinetics of formation and also during the subsequent metal conversion stage of the two compounds. A series of experiments were conducted by varying pH, solution concentration and temperature of precipitation and the findings are discussed in the following sequel. In each experiment, 100 ml of synthetic cobalt chloride solution was used for precipitating ammonium cobalt fluoride with a known amount of ammonium fluoride solution. The compound resulted was mainly identified by XRD analysis. The residual cobalt in the filtrate is the indicator of the reaction yield.

# 3.1.1. Influence of strength of ammonium fluoride solution

Ammonium fluoride concentration was varied from stoichiometric requirement to 110% excess, at pH 5, and a minimum temperature of 60 °C for every 100 ml of 1 M cobalt chloride solution. The results are presented in Table 1. The weight of the product formed was very much influenced by the quantity of the precipitating agent. Almost 100% excess of ammonium fluoride was found necessary for complete precipitation. The experiments also indicated the product to be highly soluble at the experimental temperature of 60 °C. The filtrate analysis (included in the same table) confirms the result. All the products were further substantiated by XRD (Table 2).

# 3.1.2. Influence of temperature

The next set of experiments were conducted, to study the effect of reaction temperature on ammonium cobalt fluoride precipitation by varying the temperature of cobalt solution from 30 to 90 °C. Table 2 summarizes the results of the experiments on precipitation at these different temperatures. As can be seen from the table, with rise in temperature, a decrease in weight of the precipitate and increase in Co value in the filtrate occurs. This can be attributed to an increase in solubility of the precipitate with increase in temperature and thereby reduces the weight of

| Table 2                                |  |
|--|--|
| Effect of temperature on precipitation |  |

.....

| S. no. | Temperature (°C) | ACF (g) | Co in filtrate (g) |
|--------|------------------|---------|--------------------|
| 1      | 30               | 24.4    | Nil                |
| 2      | 50               | 20.5    | 1.9                |
| 3      | 60               | 19.6    | 1.92               |
| 4      | 70               | 18.0    | 2.60               |
| 5      | 80               | 17.8    | 2.75               |
| 6      | 90               | 17.5    | 2.80               |

Co = 1 M (100 ml), pH 5, NH<sub>4</sub>F = 100% excess.



Fig. 4. X-ray diffraction analysis showing peaks for NH<sub>4</sub>CoF<sub>3</sub>.



Fig. 5. XRD data depicting the peaks for  $(NH_4)_2CoF_4$ .

the precipitate formed. To reduce solubility, an optimum temperature of 30 °C was chosen for further experiments which also resulted in maximum amount of ammonium cobalt fluoride precipitate. XRD pattern of the precipitate formed at room temperature and 50 °C is shown in Figs. 4 and 5. It is evident from Fig. 4 that precipitation reaction at room temperature (i.e. 30 °C) results in the formation of only NH<sub>4</sub>CoF<sub>3</sub> phase. Any further increase in temperature leads to formation of (NH<sub>4</sub>)<sub>2</sub>CoF<sub>4</sub> as seen in Fig. 5.

# 3.1.3. Influence of pH on precipitation

Table 3 summarises the results of studies conducted by varying pH of precipitation of cobalt. In this set of experiments, a known amount of ammonium fluoride was added to 1 M cobalt

Table 3 Effect of pH on ammonium cobalt fluoride precipitation

| S. no. | pH | ACF (g) | Co in filtrate (g) |
|--------|----|---------|--------------------|
| 1      | 2  | 18.4    | 2.45               |
| 2      | 3  | 19.0    | 2.10               |
| 3      | 4  | 19.8    | 1.80               |
| 4      | 5  | 24.0    | Nil                |
| 5      | 6  | 23.0    | 0.20               |

Co Conc = 1 M (100 ml),  $NH_4F = 100\%$  excess, temperature =  $30 \circ C$ .

| Table 4             |               |              |              |          |     |
|---------------------|---------------|--------------|--------------|----------|-----|
| Results of study on | thermal decom | position tem | perature and | time for | ACF |

| S. no. | Temperature (°C) | Time (h) | Remarks                     |
|--------|------------------|----------|-----------------------------|
| 1      | 400              | 6        | Incomplete red <sup>n</sup> |
| 2      | 450              | 6        | Incomplete red <sup>n</sup> |
| 3      | 550              | 6        | Completely reduced to metal |
| 4      | 650              | 6        | Agglomerated powder         |
| 5      | 550              | 4        | Incomplete red <sup>n</sup> |
| 6      | 550              | 5        | Incomplete red <sup>n</sup> |
| 7      | 550              | 6        | Complete red <sup>n</sup>   |
| 8      | 550              | 7        | Agglomerated powder         |

chloride solution. The pH of the solution was varied from 2 to 6, at a constant temperature of  $30 \,^{\circ}$ C with stirring. The precipitate formed depends very much on the initial pH of the solution. The precipitation of ammonium cobalt fluoride does not occur at once, on attaining the desired temperature, but later, after an induction time of stirring for half an hour. The precipitate obtained was filtered without washing and air dried for 6 h. The product was weighed and filtrate was analysed for calculating the yield. As seen from the table, condition of precipitation stabilizes at around pH 5 with maximum precipitate and minimum loss of cobalt in the filtrate. At acidic pH, Co shows a tendency of solubility while higher pH warrants the formation of hydroxide. Hence, an optimum pH of 5 was chosen for all further experiments.

# *3.2. Decomposition of ammonium cobalt fluoride to cobalt metal*

The dried precipitate of ammonium cobalt fluoride was decomposed in a closed reactor, in argon atmosphere, under a positive pressure of around  $(1-1.5) \times 10^5$  Pa. Optimum condition of decomposition was studied in detail by varying the temperature and time of the reaction and the findings are presented in Table 4. From the results it was ascertained that an optimum temperature of 550 °C was essential. Temperatures lesser than 550 °C resulted in incomplete reduction, with sizable amount of the intermediate product, cobalt fluoride, along with partially reduced cobalt metal. Higher temperatures resulted in agglomeration of the product with an increased particle size.

However, the decomposition had to be coupled with an optimum duration also. Hence, by applying the optimum temperature (550  $^{\circ}$ C), time of heating was varied from 4 to 7 h. A minimum period of 6 h was adequate for complete conversion of cobalt ammonium fluoride to cobalt metal. Longer duration of reduction results in a tendency towards sintering of the product. All the experiments were backed by XRD analysis to identify and characterise the product.

#### 3.3. XRD analysis

XRD investigations conducted to identify the products at intermediate and final stages of the process scheme are presented in Figs. 4–7. Figs. 4 and 5 indicate the formation of compounds  $NH_4CoF_3$  and  $(NH_4)_2CoF_4$  at different conditions of precipitations. It is evident from these two figures that the temperature



Fig. 6. XRD analysis depicting incomplete reduction of (NH<sub>4</sub>)<sub>2</sub>CoF<sub>4</sub>.

play a decisive role in the formation of a particular ammonium cobalt fluoride compound. The decomposition products of these compounds were analysed from Figs. 6 and 7. As seen from the XRD data, the compound NH<sub>4</sub>CoF<sub>3</sub> formed at room temperature precipitation and further decomposition at  $550 \,^{\circ}$ C resulted in pure cobalt powder while (NH<sub>4</sub>)<sub>2</sub>CoF<sub>4</sub> (formed at a higher temperature) on decomposition at a temperature of  $550 \,^{\circ}$ C results in incomplete decomposition and a mixture of the products (Fig. 7), perhaps due to the following reaction [13]:

$$(NH_4)_2CoF_4 \rightarrow NH_4CoF_3 + HF$$
 (7)

which on further heating results in CoF<sub>2</sub>.

It was observed that, the additional step in the reaction requires either longer duration of heating or still higher temperatures for complete conversion to metallic powder. However, these experiments were not attempted in detail, since maximum conversion to cobalt metal powder was obtained from  $NH_4CoF_3$  precipitate obtained at room temperature. Hence, room temperature precipitation of 1 M CoCl<sub>2</sub> at a pH 5 and subsequent decomposition at 550 °C in an air tight reactor, for a duration of 6 h was identified as optimum experimental condition for the process. It may be stressed here that, the process was devoid of any external use of hydrogen.

The cobalt powder obtained was of high purity, nonpyrophoric and of very fine size. In many cases the pyrophorocity or inflammability of the powder of a given material will vary with



Fig. 7. X-ray diffraction analysis of cobalt powder resulted from decomposition of NH<sub>4</sub>CoF<sub>3</sub>.

| Table 5                                   |  |
|---|--|
| Chemical analysis (wt.%) of cobalt powder |  |

|    | · · · · |
|----|---------|
| Со | 99.95   |
| Ni | 0.04    |
| Cu | 0.003   |
| Fe | 0.01    |
| Mg | 0.003   |
|    |         |

the process used to produce it. It is because either the surface per unit weight of powder will vary or chemical reactivity of the material itself will differ [14]. As far as the pyrophorocity of the powder is concerned, the activity of a powder refers to its propensity to react with the environment; and it depends on the rate of material transport by bulk and surface diffusion, the rate of adsorption and desorption, and similar reactions with the environment. The average particle size or diameter of a powder is one of the several factors which determines its activity. Small or finely divided particles are usually, but not always of greater activity than larger particles. With decreasing particle size of a powder, the behavior of the particle is governed increasingly by surface phenomena. The variables such as, interparticle porosity, oxide films, solid impurities, adsorbed gases or moisture, etc. have a pronounced effect on pyrophoric tendencies [15]. In this case, the SEM analysis of the powder revealed it to be of rounded/spherical shape, often loosely agglomerated powder with a minimum interparticle porosity. The minimum porosity decreases the chances of oxygen adsorption which in turn reduces the inflammability of the particles.

The purity of the cobalt powder was determined by atomic emission spectroscopy and induction coupled plasma techniques and its result is indicated in Table 5. Since the cobalt powder was prepared from analytical grade cobalt chloride salts, the ultimate cobalt powder was 99.95% pure.

#### 3.4. Scanning electron microscopy

Cobalt powder was charecterised with respect to size and morphology by scanning electron microscope (SEM). Though a detailed investigation of all the powder is beyond the scope of this paper, a few images of the Co powder are depicted in Fig. 8a-c, respectively. The effect of cobalt concentration on the morphology of the powder can be demonstrated with these SEM micrographs. As shown in figures, there is a significant change in the size of cobalt powders formed by varying the concentration of cobalt chloride solutions. Powders formed from very dilute solutions such as 0.25 M results in very fine cobalt powders of particle size in the range of 100-250 nm (Fig. 8a). While particle size increases from 500 nm to 1.1 µm by increasing the strength of cobalt solutions from 1 to 3 M (Fig. 8b and c). As mentioned earlier in Table 4 (result 4 and 8) the cobalt metal powder formed at temperature of 650 °C and by heating for longer durations were agglomerated. The SEM analysis of these powders recorded slightly larger sizes, in the range of  $1.1-1.5 \,\mu\text{m}$ . It is clearly evident from all the micrographs that powders produced by these process are spherical/round in morphology and loosely bound which qualifies it to be suitable for binder in cemented carbide tools.



5 um

DET: SE Detector

Device: VEGA MV2300T/40





SEM MAG: 20.00 kx WD: 8.0953 mm VAC: HiVac

(a)

DET: SE Detector 2 µm DET: SE Detector 2 µm Device: VEGA MV2300T/40

Vega ©Tescan Digital Microscopy Imaging Sem\_zero\_point\_25\_Mag\_20K Shovit



WD: 13.8169 mm

VAC: HiVac

(b)

Fig. 8. (a) SEM of Co powder from  $0.25 \text{ M CoCl}_2$  (100–250 nm); magnification:  $20\text{K}\times$ ; (b) SEM of Co powder from 1 M CoCl}2 (300–500 nm), magnification:  $10\text{K}\times$ ; (c) SEM of powder from 3 M CoCl}2 (450–1100 nm), magnification:  $10\text{K}\times$ .

# 4. Conclusions

An alternate method to form extra fine cobalt powder from chloride solution via ammonium cobalt fluoride intermediate was test examined. Optimum parameters were established for the precipitation of an appropriate ammonium cobalt fluoride. The presence of an additional compound of ammonium cobalt fluoride deteriorate the quality of powder and yield of the process. Complete decomposition parameters of ammonium, cobalt fluoride to cobalt powder were also standardized. Cobalt metal powder prepared by this process does not involve external supply of hydrogen and neither got oxidized nor caught fire during handling and storage in spite of the extra fine size. The SEM analysis of the powder revealed it to be of rounded/spherical shape, often loosely agglomerated powder with a minimum interparticle porosity.

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