

## Synthesis of WC–Ni composite powders by thermochemical processing method based on co-precipitation



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### HIGHLIGHTS

- Homogeneous mixtures of Ni and W oxides were prepared by chemical co-precipitation.
- In the precursor solution, pH ~ 13 was found to maximize the extraction of Ni and W.
- Simultaneous reduction and carburization was performed by CO to produce WC–Ni.
- The reactions and mechanism of precipitation were elicited.

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### ABSTRACT

The synthesis of nano-sized, uniformly distributed WC–Ni powders was studied through co-precipitation of oxide powders followed by thermochemical reduction and carburization. Ni and W oxide complexes were first obtained from their salts' solutions through chemical co-precipitation method by adjusting pH. The precipitated powders were then calcined to obtain a mixture of their oxides as WO<sub>3</sub> and NiWO<sub>4</sub> which were reduced and carburized simultaneously by carbon monoxide (CO) at temperatures up to 950 °C. ICP and FTIR along with XRD analyses were conducted on the residual solution and powders to investigate the precipitation mechanism. The effect of precursor solution pH on the chemical and morphological uniformity of the powders process was investigated; the optimum value was determined as ~13. The ICP analysis of the remained solution after co-precipitation indicated almost complete extraction of Ni and W into the precipitates. Proper temperatures for calcination in air, and simultaneous reduction and carburizing in CO were determined through TGA analysis; 550 °C and 950 °C respectively. SEM observation of the material in various steps revealed nano-sized particles in all of them, along with fine distribution of Ni and W elements. XRD analysis of the calcine sample showed the presence of WO<sub>3</sub> and NiWO<sub>4</sub> and the same analysis for the reduced and carburized sample confirmed the formation of WC and Ni.

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### 1. Introduction

WC based cemented carbides have a leading position in industrial applications requiring good wear resistance, particularly abrasive wear resistance [1,2] such as cutting-, drilling-, and forming-tool materials [3,4]. This is because of the unique properties of WC including high hardness, a low coefficient of thermal expansion, a certain amount of plasticity, and good wettability by

molten metal [2,5]. The brittleness of the carbide phase is moderated by embedding the particles in the metal matrices (more ductile phases) like Co, Ni, Fe, etc. [2]. Among different binders, cobalt and nickel have been commonly used due to its excellent wetting and adhesion to WC, as well as adequate mechanical properties. Nickel has higher corrosion resistance than cobalt and thus has improved wear resistance in aggressive environments [1,2,4,6–8] although the WC–Ni cemented carbides are of inferior mechanical properties compared to WC–Co based materials [9]. The conventional synthesis methods for WC–Ni cemented carbides involves the use of mechanical routes (powder metallurgy) to grind and homogenize the components [2,4,6,10–13] which have significant disadvantages of the high energy requirement, loss of the

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fine grain structure and, contamination of the material by milling environment and tools [14,15]. Due to the density differences between WC and Ni, providing a homogeneous mixture from these materials through the milling process seems to be a genuine challenge. It has proven to be difficult to obtain a uniform particle size distribution and chemical dispersion (a homogeneous elemental distribution of W and Ni). Recently, thermochemical process has been employed as a promising novel method to address these problems. Based on this method, two different salt precursors are utilized to produce the oxides. After calcinations, the oxide powders are subjected to reduction by hydrogen, in order to obtain oxygen free bi-metal (in this case W–Ni) powders [16–18]. Further, the synthesized composite powders should be ultimately carburized to generate WC from elemental tungsten which is possible by a carburizing agent [19]. The co-precipitation method has been applied for the systems of W–Cu and W–Co successfully [14,15,20,21] due to the rich solution chemistry of tungsten (VI) which can form both stable and, due to slow kinetics, metastable species. Also, Mohamed et al. [22] have reported synthesis of nanocrystalline NiWO<sub>4</sub> by co-precipitation and sol–gel techniques using polymeric templates of polyethylene glycol and triblock copolymer respectively. Using this method, one can make sure about the feasibility of generating composite powders with fine particles and a narrow size distribution. Moreover, a very high level of chemical dispersion is achieved from utilizing homogenous precursors containing ions of W and Ni mixed on the atomic scale. This can have beneficial impacts for the mechanical and corrosive properties and sinterability of the ceramic–metal mixture due to the fine and uniform particle size [23–26]. The present study aimed at synthesizing WC-10 wt.% Ni submicron powders by chemical route and produce a homogeneously dispersed powder with a narrow size range. Understanding and optimizing the precipitation process to achieve this was an objective of the study as little is known for the W–Ni system in that regard. Further, the possibility of carrying out both reduction and carburization in one step, using CO was studied as an alternative to a two–step method conventionally employed.

## 2. Experimental procedures

The precursors of W and Ni were aqueous solutions which were prepared by separately dissolving 33.661 g of Na<sub>2</sub>WO<sub>4</sub>·2H<sub>2</sub>O (>99.0% purity, Merck, Germany) and 9.608 g of NiSO<sub>4</sub>·6H<sub>2</sub>O (99.0% purity, Merck, Germany) salts in the 200 mL and 20 mL distilled water respectively. Concentration of Ni and W ions was based on the stoichiometric proportions to produce a final WC-10 wt.% Ni powder. To adjust the pH of the Ni solution to a base, ammonia solution was added. The two precursor solutions were then mixed

together and heated on a stirrer hot plate at 80–90 °C for 5 h in order to have desirable complexes precipitated. The precipitate was separated from the solution by filtering and the remained solution was analyzed by ICP-OES (Jobin Yvon 138) to determine any residual reactant elements. The separated precipitates were rinsed with water for several times and then dried at 110 °C for 4 h. In order to determine the appropriate calcination temperature, the dried sample was subjected to thermogravimetry analysis (TGA) (SETARAM 92-16.18) up to 750 °C with a heating rate of 10 °C/min in air. Then the dried sample was calcined in an electrical muffle furnace at 550 °C for 2 h in air to have oxide powder. The temperature for complete reduction and carburizing of oxide phases in CO atmosphere was determined by heating sample at the rate of 10 °C/min under CO flow rate of 50 mL/min. Heating to and cooling from this temperature were carried out under Ar flow, at the rate of 10 °C/min. The products were analyzed by XRD (PW1800 Philips, Cu- K<sub>α</sub>) to identify the present phases. The microstructure and morphology of the dried, calcined and carburized powders and also their homogeneity were studied by scanning electron microscope (FEI Quanta FEG 250 Environmental SEM/STEM). Tungsten and nickel distribution of material in different stages were investigated by elemental mapping using EDS.

## 3. Results and discussion

The color of the precursor salts of W and Ni were white and emerald green respectively. The nickel salt NiSO<sub>4</sub>·6H<sub>2</sub>O is dissolved in water by ionizing to Ni<sup>+2</sup> and SO<sub>4</sub><sup>-2</sup> resulting in an acidic solution. The nickel ions form a hydroxide complex by accepting H<sub>2</sub>O molecules according to Reaction 1 [27]:



Transition metal ions combine easily with neutral molecules or anions as ligands to form coordination complexes. The number of ligands that bind to a metal center usually varies. Most complexes have the coordination number of 6 in which the ligands are arranged around the metal center in octahedral geometry. The nickel complex exhibits the usual, very symmetrical, octahedral geometry. Nickel has an electron structure of Ar4s<sup>2</sup>3d<sup>8</sup> and the electron structure of its cation is Ar4s<sup>0</sup>3d<sup>8</sup> so it has empty orbitals in 3d, 4s and 4p. Active lone pairs of electrons in the outer energy level of H<sub>2</sub>O molecules are used to form co-ordinate bonds with the nickel ion as 6 ligands (lone pair donors) around Ni<sup>+2</sup> ions (Fig. 1) to form hydrolyzed nickel. This complex has light green color. Tungsten salt solution is colorless and it forms Na<sup>+1</sup> and WO<sub>4</sub><sup>-2</sup> ions on dissolution, so the solution is alkaline as pH measurements confirm (pH = 9).

An attempt was made to determine the role of pH in forming components during co-precipitation by mixing solutions of initial

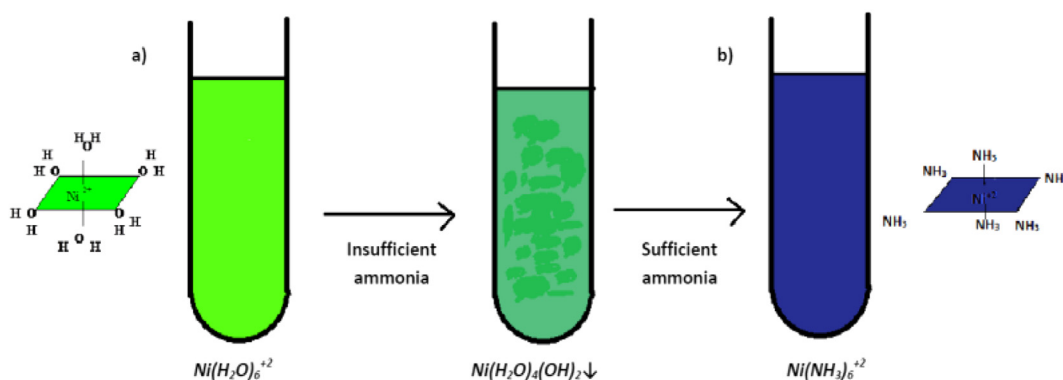


Fig. 1. The complexes of Ni<sup>+2+</sup> with ligands of a) H<sub>2</sub>O molecules and b) NH<sub>3</sub> molecules.

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