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# Characteristics of WC Nanopowders with Process Atmospheres of Chemical Vapor Condensation Process

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## 1. Introduction

Researches on nanostructured materials with grain size of 1~100nm range have been intensively carried out in recent decades, because it has unique properties, which are different from that of conventional coarse-structured materials, such as high catalysis and electromagnetic properties and high mechanical performance[1].

The nanostructured powders have been fabricated by various processes such as inert gas condensation process(IGC), mechanical alloying(MA), mechano-chemical process(MCP), and chemical vapor condensation(CVC). Among them, the CVC process is very attractive method because it can produce the nanostructured powders with high purity and non-agglomeration properties as well as grain size of <30nm[2]. CVC process has an advantage applicable to almost all materials because wide metal-organic precursors are available and it can also produce a large amount of nanopowders. The size of CVCed powders are mainly affected by reaction temperatures, and the phase and composition are dependent on as carrier gases or reaction atmospheres.

In this study, we synthesized tungsten carbide(WC) and tungsten(W)-based nanopowders by chemical vapor condensation process by using of metal-organic precursors. Effects of the carrier gases and reaction atmospheres on the phase, microstructure and size of as-synthesized nanopowders were investigated.

## 2. Experimental Procedure

The CVC apparatus are similar with hot-wall reactor type furnace. Basic setup of the CVC adapted in this experimental is shown in Fig 1. Vaporization temperatures were optimized at 120℃. High-purity carrier gases fed through a heated bubbling unit containing the solid tungsten hexacarbonyl($W(CO)_6$) precursor. Three different carrier gases such as CO, Ar and $O_2$ of 1atm. was used to get different the W-based nanosized powders. Nanopowders was also produced under the vacuum of $1\times10^{-4}$ atm. The CVC experiment was conducted at temperature range of 500~1000°C.

X-ray diffraction(XRD) with monochromatic CuKa radiation was performed to identify the phases existing in as-prepared powders. Chemical composite of CVCed powders was examined by ICP-MS. The powder size was calculated by Scherrer equation from the half width of XRD peaks and BET specific surface area. The morphology and powder size were also analyzed by field-emission scanning electron microscope(FE-SEM, Philips) and transmission electron microscope(JEM-2000FXII). The powders for TEM investigations were ultrasonically dispersed in ethanol and dropped on a carbon-coated copper grid.

## 3. Results

The hexacarbonyl tungsten ($W(CO_6)$) was decomposed from $W(CO_6)(s)$ to $W(g) + 6CO(g)$ in the temperature range of 150~205℃. In order to investigate the phase changes with carrier gases, the as-CVCed powders, which were collected in the chamber, were analyzed by XRD as shown in Fig. 2 XRD results show that cubic-tungsten carbide($WC_{1-x}$), pure tungsten(W) and monoclinic W-based nanopowders were synthesized under CO, Ar and $O_2$ carrier gases, respectively. However, XRD peak of the CVCed powders was greatly broadened in the vacuum atmosphere. As the reaction temperature increased from 600℃ to 1000℃, the size of CVCed $WC_{1-x}$ powders decreased from 53nm to 28nm. The powder phase of cubic-$WC_{1-x}$ was not changed up to 800℃, but new peaks, which corresponded to pure tungsten phase, was confirmed at 1000℃. Grains size of the $WO_3$ powders increased with increasing of temperature form 10nm to 38nm.

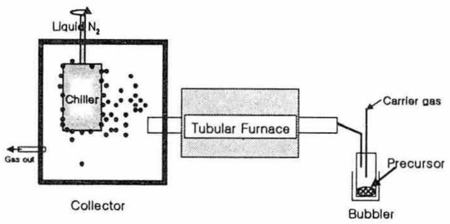


Figure 1. Schematic of CVC process.

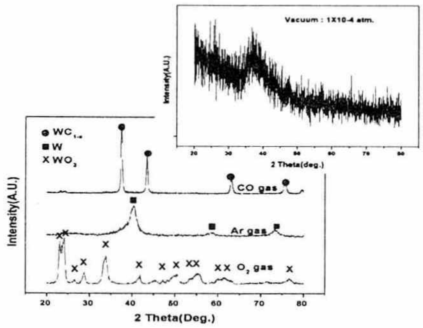


Fig. 2. XRD traces of the CVCed powders under different carrier gases(at 700℃)

TEM observation showed that the CVCed tungsten carbide powders had loose aggregates that consisted of point contacts of powders. The CVCed $WC_{1-x}$ powders had very smooth surface morphology and rounded cubic shape. It was reported that monoclinic $WO_3$ nanorods or nanobelts could be synthesized via physical vapor deposition process at 950-100℃ in air[3]. However, in this study, the as-prepared $WO_3$ nanopowders showed very smooth spherical shape in FE-SEM and TEM micrographs. No $WO_3$ nanorods or nanobelts were visual.

## 4. Conclusions

Nanostructured W-based nanopowders were prepared by chemical vapor condensation process by using of hexacarbonyl tungsten ($W(CO)_6$). Cubic $WC_{1-x}$, pure W and monoclinic $WO_3$ nanopowders were synthesized with changes of the carrier gases. Grain size of $WC_{1-x}$ powder was decreased with increasing of reaction temperature, but $WO_3$ powder size increased.

## Acknowledgement

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## References

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