Synthesis of Ultrafine TaC Powders by Carbothermal Reduction

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1. Introduction

Tantalum carbides based materials have very high melting points(>3500℃) and a high resistance to chemical attack or thermal shock, which makes them useful for high temperature and high stress engineering applications.

The addition of TaC to WC-Co alloy improve the performance in machining steels by reducing front rake wear, creating, and the welding tendency between tool and chip, and inhibit the recrystallization of the carbide phase, and therefore the resulting alloys have generally a finer grain and higher hardness than corresponding TaC-free compositions. Such alloys are also characterized by a wider sintering range and thus are less sensitive to oversintering.

These carbide powders have previously been produced by direct reaction of tantalum metal with carbon, gas phase reaction of $TaCl_5$ with hydrocarbon, or solid state reaction of Ta_2O_5 with carbon. It has been reported that formation of the carbides by the solid-state reaction proceeds at high temperature(>1400℃) under high vacuum, but higher temperatures(>1500℃) are necessary for complete reaction.

The objective of this work is to synthesize ultrafine TaC powders by means of carbothermal reduction using tantalum oxalate solution as raw material and spray drying process.

2. Experimetal

The precursor solution for spray drying was prepared from a mixture of 621.6㎖ tantalum oxalate solution(H. C. Starck, Germany) and 5000㎖ deionized water

The solution was dried to form a precursor powders by means of spray-drying. The conditions of spray drying were set as follows : temperature of intake air 250℃, outlet air 130℃, atomiser rotation speed 11000 rpm, and solution feed 20㎖/min.

Powders obtained after spray drying were directly calcined in air at different temperatures from 500℃ to 700℃ for 2 hours, and then this powders calcined at 500℃ were crushed and mixed with carbon black by wet ball milling. The milling was performed in a vessel with WC-6wt.%Co balls of 6㎜ diameter in n-hexan for 24hours. The ball/powder weight ratio was 40:1.

The thermal decomposition behaviour of the precursor powders was examined by thermogravimetric analysis (TGA/DTA, TA Instruments, SDT2960). X-ray diffraction (XRD, Cu Kα, Rigaku Co., D/max-2200) were used to analyze the crystalline phases of the spray dried precursor, the calcined powders and carburized powders. The morphology and microsructures of the powders were examined by means of field emission-scanning electron microscopy (FE-SEM, Phillps Co., X130 SFEG) and transmission electron microscopy(TEM, JEOL Co., JEM-2000FX II). The crystallite size of TaC powders was calculated by Scherrer's formula from XRD peaks.

3. Results and discussion.

The spray dried powders consist of smooth spherical particles approximately under 30㎛ in diameter and have amorphous structures.

The powder calcined at 500°C and 600℃ are primarily amorphous in structure, as shown by a broad continuum in XRD pattern. when the precursor powder is heat-treated at 700°C for 2 hours, the Ta_2O_5 phase is formed.

The weight loss for spray dried powders occurs in two steps. The weight loss in the range from room temperature to 500℃ can be assigned to the evoltion of H_2O, CO, CO_2. The second weight loss step occurs in the range of 700-750℃ corresponding to the elimination of CO, CO_2. The final decomposed product corresponds to the Ta_2O_5.

The TEM micrographs show that calcined powders are small spherical grains with a narrow size range. The average particle sizes of powders calcined at 500℃ and 700℃ are under 50 nm. The average particle size increased as the calcining temperature increased.

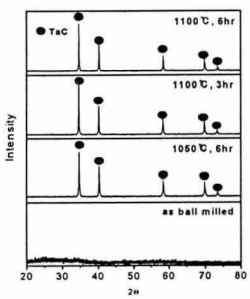

Fig. 1 shows XRD results for materials synthesized from reaction of amorphous tantalum oxide and carbon. It is confirmed that the carbide reacted at 1050℃ and 1100℃ are composed of single phase, TaC. There are no peaks corresponding to tantalum, tantalum oxide, Ta_2C.

Fig. 1. XRD patterns of the carburized powders with reaction temperatures and times.

Fig. 2 shows the TEM images of TaC powders after carburization. There are a few agglomerates among the particles, and the particle size distribution is nonuniform, the average particle size is about less than 200 nm in diameter.

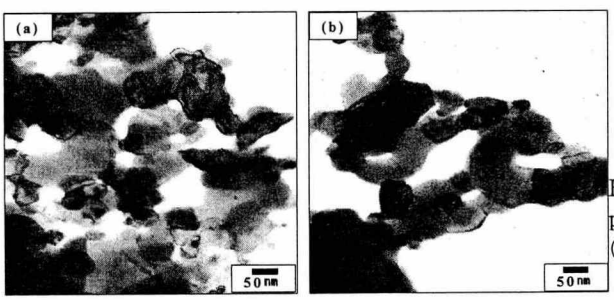

Fig. 2. TEM micrographs of the powders carburized at (a) 1050℃ and (b) 1100℃ for 6hours

The size of TaC crystallites has been estimated from the full-width at half maximum (FWHM) of the (111) diffraction peak using Scherrer's equation. The average crystallite size works out to be about 50nm.

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