1. Introduction

New method of welding process is Friction Stir Welding method (FSW) that gets the limelight. FSW was developed in 1991 and is successfully being applied to an increasing number of joining applications worldwide. The FSW advantages of this solid-state joining process, which uses frictional heat generated by a rotating and traversing cylindrical tool with a profiled pin along a square butt weld joint, encompass better mechanical properties, low residual stress and deformation, weight-savings, and reduced occurrence of defects, compared to the conventional welding methods. Recently, research on the FSW process has mainly focused on the joining of low-melting metallic materials, such as aluminum alloys with steel. Tungsten carbide-cobalt hard materials (WC-Co) are widely used for a variety of welding tool, machining, cutting, drilling, and other applications. Morphologically, they consist of a high volume fraction of the “hard” hexagonal WC phase embedded within a soft and tough Co binder phase. An increase in the density of WC-6wt.%Co was not accomplished by conventional sintering method. New sintering method of Spark Plasma Sintering (SPS) was utilized to consolidate WC-xwt.%Co hard materials using various weight percent of Co powders. The demonstrated advantages of these processes are rapidly densification to near theoretical density in a relatively short time and without significant change in a grain size.

2. Experimental Procedure

In this study, 99.95% pure tungsten carbide (0.4–0.5 μm) and 99.8% pure cobalt (1.6 μm) powders were used as the starting materials. A particle size analyzer was used to examine raw WC and Co materials (Malvern, mastersizer 2000E). The WC and Co particle sizes were 0.2–0.3 μm and 10–20 μm, respectively. Additionally, the average grain sizes of WC and Co were about 0.3 μm and 15 μm with distributions of d(0.1) : 0.129 μm, d(0.5) : 0.199 μm, d(0.9) : 0.384 μm and d(0.1) : 6.413 μm, d(0.5) : 12.098 μm, d(0.9) : 23.844 μm, respectively. The mixed WC and Co powder with a molar ratio of 94:6 was milled in a high energy ball mill (planetary mill) at 250 rpm for 10 h. Tungsten carbide balls (6 mm in diameter) were used in a sealed cylindrical stainless steel vial with alcohol. The weight ratio of the balls-to-powder was 10:1, and the alcohol-to-powder was 2:1. The milling significantly reduced the grain size of the powder. The grain size and the internal strain were calculated using Stokes and Wilson’s formula,
\[ b = b_d + b_e = k \lambda / (dcos \theta) + 4 \epsilon tan \theta \]

where, \( b \) is the full width at half-maximum (FWHM) of the diffraction peak after the instrument correction; \( b_d \) and \( b_e \) are the FWHM for a small grain size and internal stress, respectively; \( k \) is a constant (with a value of 0.9); \( \lambda \) is the wavelength of the X-ray radiation; \( d \) and \( \epsilon \) are the grain size and the internal stress, respectively; and \( \theta \) is the Bragg angle.

The parameters \( b \) and \( b_s \) follow Cauchy’s form with the relationship: \( B_0 = b + b_s \), where \( B_0 \) and \( b_s \) are the FWHM of the broadened Bragg peaks and the standard sample’s Bragg peaks, respectively. Fig. 1 shows the XRD patterns of the raw powders and the milled WC-6wt.% Co powder mixture. The FWHM of the milled powder was greater than the raw powders because of the reductions in the internal strain and the grain size. The average grain sizes of the milled WC and Co powders were 278 and 33 nm, respectively. Additionally, the milled powder exhibited a particle size of about 100 nm and appeared to have a distribution of \( d(0.1) : 0.062 \ \mu m, d(0.5) : 0.094 \ \mu m, d(0.9) : 0.141 \ \mu m \). After milling, the mixed powders were placed in a graphite die (outside diameter, 30 mm; inside diameter, 10 mm; height, 40 mm), and then placed into a Spark Plasma Sintering system that was made by Sumitomo Coal Mining in Japan. The heating rate was approximately 100 °C/min during this process. At the end of the process, the current was turned off, and the sample was allowed to cool to room temperature. The relative densities of the sintered samples were measured using the Archimedes method. The microstructural information was obtained from the product samples, which were polished and etched using a Murakami’s reagent (5 g Fe3(CN)6, 5 g NaOH, and 50 ml distilled water), for 1 min at room temperature. The compositional and microstructural analyses of the products were carried out through X-ray diffraction (XRD) and field-emission scanning electron microscopy (FE-SEM). The Vickers hardness was measured by performing indentation tests at a load of 30 kg and a dwell time of 15 s.

3. Results

The microstructure of WC-6wt% Co nano-composite prepared by high energy ball milling was investigated by X-ray diffraction, Field Emission Scanning Electron Microscopy and Grain size analysis. Particle sizes of milled WC and Co powder were 189 and 33 nm, respectively. The sintering behavior of WC-Co powder and mechanical properties of its sintered body were investigated in this study. Grain sizes of WC and Co after sintering were 271 and 20.6 nm, respectively. Also, it was found that the Vickers hardness and fracture toughness of WC-Co sintered body were 1755.81 kg/mm² and 25.62 MPa m½, respectively.

Fig. 1. XRD patterns of WC-6wt.% Co Hard Materials; a) WC, b) Co, c) ball milled and d) after sintering