PM2010 Powder Metallurgy World Congress & Exhibition
10th – 14th October 2010
Florence, Italy

Title of Presentation: Advantages of a wet process for the production of Ti matrix composite reinforced with carbon nano materials by powder metallurgy route

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Purpose of Presentation

1. To promote the new technique of the powder metallurgy processing, namely “Wet Process”, using the combinations of chemical technique and powder metallurgy knowledge.
2. To gain an experience and learn the knowledge of powder metallurgy techniques from the various works at the conference site.

Summary of Presentation:

A titanium matrix composite reinforced with different carbon nano materials i.e. carbon nanotubes, acetylene black and carbon black was fabricated by spark plasma sintering (SPS) and hot extrusion. Commercially fine Ti powders (95% purity, TOHO TITANIUM Co., Ltd. Japan) with mean particle size of 30 μm were used as starting materials. The different kinds of carbon nano materials i.e. carbon nanotubes (CNT), acetylene black and carbon black were employed as the reinforcing materials. The CNT dimension was ~ 20 nm in diameter and 1–10 μm in length. The laser particle size measurement of acetylene black and carbon black revealed the particle size of 398 and 290 nm, respectively. A zwitterionic surfactant solution containing 0.5wt.% of those carbon nano materials was prepared. Fine Ti powders were immersed into the carbon/zwitterionic solution, and subsequently dried in an oven at 100°C for 180 min. The coated Ti powders were debinded in a horizontal tube furnace at 600 °C for 60 min. under an Ar gas atmosphere. The Ti powders with different carbon nano materials were consolidated using the spark plasma sintering (SPS) process at 800°C for 30 min. under vacuum atmosphere. The sintered Ti composite billets were heated to 1000 °C for 3 min. under a protective Ar atmosphere, and immediately underwent to the hot extrusion process. The extrusion ratio, speed and die temperature were 37, 3.0 mm/s and 400 °C, respectively. The phase characterization and microstructure of extruded Ti composites were investigated by X-ray diffractometer and a scanning electron microscope. The extruded Ti composites were machined into tensile specimen bars of 3.0 mm. in diameter and 20 mm. in gauge length. The mechanical properties of extruded Ti composites were evaluated using universal test machine under a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$.

The coating via wet processing showed a good distribution of carbon nano materials on the Ti powder surface, compared to Ti powders mixed with the bundled CNTs via the conventional ball milling process. The distribution of carbon nano materials primarily consisted of local deposition on the curve or facet of the Ti powder surfaces. It was expected

that the deposition of each carbon nano materials on the Ti powder surface depends on the shape and size themselves and the characteristic of Ti powders. A carbon content analysis of the extruded materials must be applied to measure this parameter. The results suggest that the amount of carbon deposited on Ti powders is a reciprocal proportion with the size of carbon nano materials. The smallest size of CNTs can be deposited on the Ti powder surfaces higher than the acetylene black and carbon black materials, respectively.

X-ray diffraction patterns revealed the *in-situ* formed TiC particles existing in the Ti matrix which were originated from CNTs, acetylene black and carbon black particles. Additionally, there is no existence of possible intermetallic compound peaks, such as TiO_2 or TiN . That means the oxidation and nitridation can not occur remarkably inside the extruded materials during the hot extrusion process in a normal ambience.

The microstructure of the extruded Ti/CNTs and Ti/carbon black composite material showed the dispersion of *in-situ* formed TiC particles throughout the Ti matrix, which could be attributed to the high distribution of carbon nano materials on the Ti powder surface in the context of wet processing. The grain size measurements of the extruded Ti/carbon nano materials indicate a Ti grain size of 5.8, 8.1 and 5.3 μm for CNTs, acetylene black and carbon black particles, respectively. Also, the TiC particle size measurement showed values of 1.9, 2.1 and 3.0 μm , respectively.

The yield stress (YS) and tensile strength (TS) values of the extruded Ti/CNTs composite material were increased by 50.7% and 24.8%, compared to the extruded Ti without any reinforcement. In the case of Ti/acetylene black, the increases in YS and TS were 41.2% and 22.5%, respectively. Furthermore, the increases in YS and TS of Ti/carbon black were remarkably elevated by 64.2% and 35.7%, respectively. Regarding the effect of Ti grain size, the results of all extruded materials almost showed the same value. Also the chemical analysis results of oxygen and nitrogen are shown in the same level. Those parameters did not significantly affect the increases of YS and TS, compared to the increases of carbon content.

A wet process, the zwitterionic surfactant solution containing CNTs, acetylene black and carbon black particles, was used to coat the Ti powders. The coated Ti powders revealed a good distribution of non-agglomerated carbon reinforcing phases on the surfaces. Consequently, the *in situ* formed TiC particles were highly distributed throughout the Ti matrix, resulting in the increased mechanical properties. Furthermore, the effect of grain size, oxygen and nitrogen contents did not significantly improve the mechanical properties of the extruded Ti composites materials.

Outcome:

1. Increasing of the new knowledge, understanding and technologies in the field of powder metallurgy which can be applied to my work.
2. Having a good discussion with other students and researchers who study in the same field.
3. Having the great opportunity to exhibit my academic research in the poster presentation session in the international conference.

The authors would like to thank the Japan Society of Powder and Powder Metallurgy (JSPM) for financial support.