Review

Powder Metallurgy Research and Development Activities at USTB

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SYNOPSIS

University of Science and Technology Beijing (USTB) is one of the key universities that are directly under the guidance of China Ministry of Education. Powder metallurgy activities have been carried out at USTB for about 50 years. Beijing Key Laboratory for Advanced Powder Metallurgy and Particulate Materials, which was founded on the base of Department of Powder Metallurgy at USTB, is a comprehensive base for PM education, research and development in China. In this paper, some of the current research activities and achievements in this laboratory are introduced, including ultra-fine grained cemented carbides made from nanometer powders, W-Cu and Mo-Cu alloys for electronic packages, SHS process and its applications, warm compaction, and powder injection molding.

KEY WORDS

overview, powder metallurgy, USTB

1 Introduction

Beijing Key Laboratory for Advanced Powder Metallurgy and Particulate Materials was founded in 2001 on the basis of Department of Powder Metallurgy at USTB under the joint supervision and support of Beijing Municipal Education Commission and Science & Technology Commission. PM education and research activities have been carried out at USTB since its foundation in 1952. And now there are about 20 faculties and staff, as well as over 40 Master and Ph. D students in the Lab, including 6 full professors and 2 associated professors. In addition to supervising research activities in the Lab, the faculties provide several courses related to PM, such as Principles and Processing of Powder Metallurgy, Composite Materials, Advanced Materials Synthesis Technology and Processing, for under-graduated and post-graduated students in The School of Materials Science and Engineering at USTB. Researches conducted in the Lab concern basically the advanced PM processing, the structure and properties of various particulate materials. Special emphases have been put on the development of net-shape forming techniques and synthesis of nanometer powder as well as metal-matrix composites. Fundings of our research projects come from a variety of sources, including National Key Basic Research (973) Program, High Tech Research and Development (863) Program, National Science

Foundation of China, National Advanced Materials Program for Defence, R&D program of Beijing Municipal and industry companies. Our research facilities which include advanced processing equipments and state-of-the-art instruments are highly regarded among universities within our country. The high-lights of research in the recent years are introduced in the following paragraphs.

2 Ultra-fine Grained WC-Based Cemented Carbides^{1,2)}

A novel processing for making ultra-fine grained cemented carbides was developed, with which high performance WC-based cemented carbides have been fabricated successfully. As one of the features of this process, a tailored nontoxic aqueous solution containing W and V was prepared, from which ultra-fine tungsten oxide powders could be synthesized through an ultrasonic spray conversion dryer. The tungsten oxide powders were in an amorphous state and with mean particle size of smaller than 50 nm. They could be reduced and carbonized at lower temperature (≤ 1200 °C) compared with those produced with traditional process. Therefore, the produced WC powders were very small and with spherical shape as seen in Fig. 1. The average particle size was only about 100 nm. In addition, because some V was added in the solution, the carbonized powder contained some VC which would act

Fig.1 SEM micrographs of nano-meter WC powders.

as a grain-growth inhibitor during sintering. This process has been evaluated in a kilogram scale.

A WC-Ni-Fe alloy system was selected for the application research of the ultra-fine tungsten carbide powder in order to save Co and reduce the materials cost. The WC powders, after being pulverized in an attrition ball mill, were mixed with nano-sized Ni (Fe) powders which were also made with the above mentioned ultrasonic spray conversion process from a solution containing Ni and Fe. The mixture was compacted and sintered in a sinter-HIPing furnace. The sintered WC-Ni-Fe cemented carbide showed full density and uniform microstructure with the WC grain size of only 300–500 nm. The transverse-rupture-strength and hardness of the alloy were 3500 Mpa and HRA93 respectively.

3 W-Cu and Mo-Cu Alloys³⁻⁶⁾

W-Cu and Mo-Cu alloys are widely used as heat-sink in electronic packages because of their low thermal expansion coefficient and high thermal conductivity. Usually they are produced with a sintering-infiltration process because it is very difficult to get fully dense W-Cu and Mo-Cu alloys with traditional press-sintering process from mixed powders due to the mutual insolubility or negligible solubility between the constituents. However, the complexity of the components which can be produced with sintering-infiltration process is very limited. A mechanical activation process was studied for W-Cu and Mo-Cu alloys in our Lab. The W or Mo powders were mechanically alloyed with Cu powders in a self-designed high-energy vibrating ball mill. At the beginning stage of milling, both the tungsten and molybdenum particles become finer rapidly, while the copper particles become sheet form. After milling for 3 hours, the particle size was reduced from about 4.8 μm to smaller than 1 μm . The fine W and Mo particles were uniformly distributed in the copper sheets and

Fig.2 SEM of Microstructure of sintered W-15%Cu (a) and Mo-38%Cu alloys (b).

Fig.3 SHS-Centrifugal Process ($\Phi 820 \times 2000 \text{ mm}$).

surrounded by copper. The milled powders were then pressed respectively into compacts with a relative density of about 65%. For W-15%Cu and Mo-38%Cu alloys, after the compacts were sintered in a hydrogen atmosphere at 1340 $^{\circ}\text{C}$ and 1230 $^{\circ}\text{C}$ respectively, the relative density could go up to 98.5%. Fig. 2 shows the microstructure of the sintered alloys. Their thermal conductivity was tested to be 182 W/m.K and 154 W/m.K respectively.

4 SHS Process and its Applications⁷⁻⁹⁾

In the late 1980s, we began to study Self-propagation High-temperature Synthesis (SHS) or Combustion Synthesis (CS). Two kinds of SHS approaches (SHS-Centrifugal process and SHS-Gravitational process) for the fabrication of ceramic-lined steel pipes have been successfully developed in our Lab. Fig. 3 and Fig. 4 show the principles of the two processes and the corresponding products respectively.

Fig.4 SHS-Gravitational process ($\Phi 10-325$ mm).

The ceramic-lined steel pipes made by SHS process displayed superior properties in wear resistance, corrosion resistance, thermal and mechanical shock resistance, which have been industrially applied to transport coal, coke, coal cinder, mineral powder, cement mortar, molten aluminum and oil-water. The service life of the ceramic lined steel pipes are several to tens times longer than those of conventional steel pipes. A great variety of standard ceramic-lined steel linear pipes, elbows, three-way or four-way pipes ($\phi 10 \sim \phi 820$ mm) can be manufactured in a large scales in China.

Recently, a new project was started in this research group to identify the feasibility to make porous Ni-Ti shape memory alloys for biomedical implanting materials and to

process nuclear fuel wastes with SHS technique. The results are very promising. The porous Ni-Ti components with a porosity of 40% ~ 72% and a compressive strength of 40 ~ 220 Mpa can be synthesized by SHS. An synthesized porous sample and its microstructure are presented in Fig. 5. In the approach to immobilize nuclear fuel wastes with perovskite (CaTiO_3), chromium oxide was selected as the

(a) sample (b) SEM of the alloy

Fig.5 Porous NiTi shape memory alloy component and its microstructure.

oxidant in the SHS reaction with Ti, CaO and TiO_2 to form perovskite. It was proved that over 35% of SrO could be immobilized in perovskite with the SHS method.

5 Warm Compaction and Enhanced Sintering of Ferrous-based Alloys¹⁰⁻¹²⁾

Warm compaction technique has been studied in this Lab since 1996. The emphases were put on the development of die-wall lubrication technique, selection of lubricants, and enhancement of sintering for ferrous-based alloys and composites. The electrostatic die-wall lubrication technique was tested for the warm compaction. The results showed that pure zinc stearate could be easily charged, while the other lubricants, including EBS wax, W-special wax and various mixtures of zinc stearate with EBS wax, could not be charged until the charging voltage reached to a critical value as seen in Fig. 6. Fig. 7 compared the densities of 316L stainless steel powder compacts pressed with conventional pressing and warm compaction at different pressure levels. Warm compaction could increase the density by 0.2–0.5 g/cm³. A master alloy (FeMoB) with low melting point was prepared and studied for enhancing

Fig. 6 Relationships between charge to mass ratio and charging voltage for various lubricants.

Fig. 7 Comparison of green density of 316L stainless steel powder compacts pressed with different methods.

Fig. 8 Examples of PIM components.

Fig. 9 Variation of density with sintering temperature.

sintering process through forming liquid phase during sintering. It was determined that the 316L stainless steel powder compacts could be sintered to the density of 98% by addition of the master alloy.

6 Powder Injection Molding¹³⁻¹⁷⁾

Powder injection molding (PIM) is a manufacturing process used in the fabrication of net- or near-net-shape components with highly controlled microstructures and properties. It can be used in the production of both metallic and non-metallic components. In our lab, several new binder systems as well as the debinding techniques have been invented and successfully used to fabricate Fe-Ni, Kovar, stainless steel, tungsten heavy alloys, cemented carbides, titanium and AlN ceramic components. Most of components have been put into market and widely used in

defense and civil sectors in China. Fig. 8 showed some of the produced components. Simulation technology for die-filling process of powder injection molding was developed based on ANSYS, with which the flow front, temperature, pressure and residue stress fields in the compacts could be calculated and shown dynamically by computer. It is very useful for guiding the mold cavity design and selection of processing parameters. In addition, chaos and fractal theory are being used to study PIM process. The enhanced sintering of tungsten heavy alloy compacts was investigated in order to make tungsten heavy alloy components with less distortion. Solid-state sintering to almost full density for W-Ni-Fe was realized with the nano-grained composite powder made by mechanical alloying as seen in Fig. 9, which showed potential for eliminating distortion caused by gravity during liquid phase sintering.

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