Preparation and Sintering of Nano-sized W-Ni -Fe Powder by Sol-Spray-Drying Process

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In this paper, Sol-spray drying method has been introduced to produce nano-meter W-Ni-Fe composite powder. This process consists of colloid solution formation, spray drying, calcinations of the amorphous precursor powder, milling and subsequent hydrogen reduction of oxide powder. This process technology facilitates good powder morphology (near spherical shape), uniform element distribution, and limits contamination.

The starting materials for synthesis of W-Ni-Fe composite powders were ammonium metatungstate (AMT), $(NH_4)_6(H_2W_{12}O_{40})\cdot 4H_2O$, nickel nitrate hexahydrate $(Ni(NO_3)_2.6H_2O$, iron nitrate $(Fe(NO_3)_3.9H_2O$, hydrochloric acid (HCl) and polyethylene glycol (PEG) were used in the synthesis.

A started colloid solution –gel containing W,Ni,Fe salts is prepared by dissolving their own solid metal salts into water and mixing them to obtain an aqueous solution of three-component salt mixture with a final composition of 90wt%W-7wt%Ni-3WT%Fe (90W-7Ni-3Fe). The weight concentration of the W.Ni,Fe salt in is 20wt% in the solution. Drops of hydrochloric acid were added into the solution mixture to acidification of ammonium metatungstate. Low molecule polymer such as PEG was added as a surface-active additive (SAA) or process control agent (PCA) to prevent particle agglomeration. Controlling the PH about 2-3 and the amount of PEG 0.6g/L formed. Spray drying was performed using a rotary atomizer with a solution feed rate of 40-50ml/min and an atomizer rotating speed of 20000-25000 rev/min in a hot air (350℃) stream, a precursor powder was obtained.The precursor obtained by spray drying was calcinated at 500℃ for 1H in air to remove organic component and form a W-Ni-Fe oxide mixture powder. The oxide mixture powder was ball milled for 1h, and was subsequently reduced in hydrogen atmosphere in the range of 500-700℃.The reduced powder was compacted and then sintered in H_2 atmosphere at1390℃ for 2h. The powder and the sintered parts were characterized by morphology, particle grain size, phases, sinterability, and microstructure through SEM, X-ray diffraction, chemical analysis and density examinations.

The precursor powder by spray drying has a spherical shell-shape with agglomerates of nano-scale sized particle with 30-50nm in particle size. The average particle size of the shell-structure agglomerates is about 3μm.

The reduced W-Ni-Fe powder at 600℃ for 2h contains a bcc tungsten (W) phase and a fcc γ-(Ni,Fe) phase. With increase of temperature, the powder has an increase in tungsten grain size. At temperature increasing above 700℃, the grain size increases dramatically.

The reduced W-Ni-Fe composite powder at 700℃ for 90min has nearly spherical morphology and uniform particle size, the average particle size is below 100nm. From differential thermal analysis (DTA) of the 90W-7Ni-3Fe composite powder, the melting point of the γ-(Ni,Fe,W) phase is 1365.8℃. In the traditional W-Ni-Fe alloys, the melting point of γ-(Ni,Fe,W) phase is about 1435℃, in comparison with traditional W-Ni-Fe alloys, the melting point of γ-(Ni,Fe,W) phase lowers to about 70℃.This means atomically/molecularly homogeneous mixing among W,Ni,Fe results in changes in powder sintering behavior. The liquid sintering temperature for densification decreases a lot, which is about 70℃. The sample sintered at 1390℃ for 120min has high density(the relative density is about 99%), good tensile properties: the tensile strength is about 900MPa, the elongation is about 13%. Sintering for shorter time cannot assure the high density and the mechanical properties. It is observed that the sintered sample at 1390℃ for 120min has spherical grains uniformly dispersed in the ductile binder phase. The average grain size is about 20-25μm.

The tensile rupture morphology shows typically transverse fracture of tungsten grains and ductile of the binder phase. Addition of small amount of Y_2O_3 earth oxide dispersion can decrease the grain size a little and increase tensile strength greatly(about 1022MPa).

The microstructure and sintering behavior of 90W-7Ni-3Fe nano-meter composite powder produced by sol-spray drying process. The spray –dried powder has spherical, hollow-structured agglomerates. The 90W-7Ni-3Fe powder consists of particle less than 100nm in size in which tungsten grain size is amounted to 35-50nm by X-ray diffraction. The powder has good sinterability, the melting point temperature of the second binder phase lowers to 70℃ than the traditional powder. The powder can be sintered to near full density (about 99%) at 1390℃, The alloy has 20-25μm and good mechanical properties : the tensile strength is about 900MPa, the elongation is about 13%. Addition of Y_2O_3 oxide dispersion can enhance the tensile strength and decrease the grain size of the alloy. The sintering behavior of the samples between MA and the spray-dried powder is different, which results in difference in sintering process, microstructure and mechanical properties.