고상연소반응법에 의한 나노텅스텐분말의 합성
Synthesis of nanometric tungsten powders by solid state combustion method

H.H. Nersisyan, 이종현, 원창환
충남대학교 급속응고신소재연구소
(haykrasom@hotmail.com)

Tungsten and tungsten heavy alloys have widespread application as radiation shielding devices and heavy duty electrical contacts. High density and good room temperature mechanical properties have generated interest in evaluating tungsten and tungsten alloys as kinetic energy penetrators against armor. Nowdays ultra fine-grained tungsten powders are in great interest because higly dense structures can be obtained at low temperature, pressure and lower sintering time. Several physical and chemical methods are available for the synthesis of nanometric metal powders: ball milling, laser abalation, vapor condensation, chemical precipitation, metallic wire explosion i.e. However production rates of the above mentioned metods are low and further efforts are needed to find out large-scale synthesis methods. From this point of view solid state combustion method (known as SHS) represents undoubted interest.

In the present work two methodologies to synthesize nanometric W under the combustion mode was developed. First is the reduction of WO_3 by magnesium in the present of NaCl as particle size controlling agent (PSCA). The formation of nanometric W powder occurs in the molten salt according to the general reaction equation:

$WO_3 + 3Mg + k\,NaCl = W + MgO + kNaCl$ (1).

Second approuch includes the reduction of WO_3 by sodium cotaining salts (NaN_3, $NaBH_4$):

$WO_3 + 6NaN_3 = W + Na_2O$ (2)

$WO_3 + 1.5NaBH_4 = W + NaBO_2$ (3).

According to thermocouple mesurement value of combustion temperature is in the interval of 850-1200 oC. After enrichment of final product single-phase W powders were allocated and examined by the technique such as XRD, TEM and BET. The results received specify of the nano-dimension of W powders. In particular the average size of the W particles received by Mg reduction method is in the range of 50-100 nm, whereas reduction by sodium salts resultes nanometric uniform W powder having 50 nm size. The values of specific surface area analyzed by BET analisis are: 5.9m^2/g at reduction by magnesium and 9-12 m^2/g by sodium containing salts.

As received powders show high sinterability in the low temperature, of particular interest 85-90 % density were obtained in the temperature of 1700-1750 °C. It is worthly to note that the sintering of tungsten powder in the industry have been performing at the temperature of 2000-2200 °C. Therefore the pellet prepared from commercional powder with the particle size 2-10 mm was not sintered in the same conditions.

The developed technology was tested in the SHS techological reactor and an oppurtunity of large-scale production was found out.