Effect of mechanical activation on optimal sintering temperature of ultrafine-grained tungsten heavy alloys

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Alloy 95wt.%W-3.5wt.%Ni-1.5wt.%Fe constitutes the target of this research. Alloys were produced using tungsten powder with average particle size of 3.9 μm, nickel powder with average particle size under 20 μm, iron powder with average particle size under 11 μm. Nanopowder compositions were obtained through mechanical activation (MA) in a planetary mill APF-3 with acceleration of grinding bodies equaling 60g (rotation rate $V_{МА}$=1450 rpm, time of mechanical activation $t_{МА}$=20 min). The alloys were sintered using the method of sintering in hydrogen (HS) and Spark Plasma Sintering (SPS).

Research into the structure of 95W-3.5Ni-1.5Fe powder composition after high-energy mechanical activation shows that the average size of tungsten particles does not exceed 100 nm. Analysis of diffraction patterns proves that with the increase in MA time, tailing of X-ray peaks corresponding to α-W is observed. With the increase in MA time, asymmetric tailing of α-W peak (110) is observed towards larger reflection angles. This indicates the formation of a supersaturated solid solution of nickel and iron in the surface layer of α-W particles.

The dependence of density on the temperature of sintering in hydrogen has two stages both for coarse-grained and mechanically activated nanopowders. Optimal sintering temperature T_1 for mechanically activated powders in hydrogen is 1300 °C, which is 150-200 °C less than the optimal tempertaure of sintering in hydrogen for coarse-grained powders (~1450-1500 °C).

Dependences of density on SPS temperature have a similar two-stage nature. The optimal SPS temperature for mechanically activated powders at the heating rate of $V_{Н}$=100 °C/min is T_1~1100 °C. Reduced heating rate leads to a shift in the optimal SPS temperature to higher values: at the heating rate of 50 °C/min the optimal SPS temperature is T_1=1200 °C. Note shall be taken that the optimal SPS temperature for coarse-grained powders is ~1300 °C.

While summarizing the outcome of experimental studies, two major results shall be taken note of: 1) decrease in the optimal sintering temperature for mechanically activated nanopowders and 2) increase in the sintering intensity of nanopowders during flash sintering. The intensity of diffusion mass transfer (I) is proportionate to the diffusion ratio D exponentially dependent on the activation energy of the diffusion process Q ($D=D_0\exp(-Q/kT)$) and to the diffusant concentration gradient (C): $I \sim -D\cdot \mathrm{grad}C$, where D_0 stands for pre-exponential factor, while k is Boltzmann constant.

It was shown that the reason for a decrease in the optimal sintering temperature of mechanically activated nanopowders is a reduction in the sintering activation energy corresponding to the grain boundary diffusion activation energy. During high-energy MA, the relaxation of stored energy takes place through the formation of grain boundaries in γ-phase particles that have an increased defect concentration. As a result, diffusion permeability of γ-phase grain boundaries after MA appears to be much higher than diffusion permeability of 'ordinary' grain boundaries in γ-phase that form as a result of sintering separate γ-phase particles with each other. Enhanced diffusion permeability found with grain boundaries of deformation origin leads to an increased intensity of tungsten atoms diffusion through γ-phase, and consequently to a decrease in optimal sintering temperature for UFG tungsten alloys.

It was shown that enhanced intensity that characterizes sintering of mechanically activated nanopowders of W-Ni-Fe system during high-speed heating occurs due to changes in the tungsten concentration gradient grad(C_w) between α-W particles and γ-phase which is a solid solution of iron and tungsten in nickel.

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