SPS of the Nanostructured Cu-Cr Composite

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Highlights

- Structure of materials obtained from Cu-Cr nanocomposite particles was investigated.
- After SPS Cu inclusions formed along the boundaries of the sintered powders.
- Two sintering schemes were used: current-assisted and current-insulated
- The structures of materials obtained by both schemes are compared

1. Introduction

The Spark Plasma Sintering (SPS) method has opened new ways for obtaining materials with different properties due to the possibility of rapid and uniform heating in conjunction with the application of pressure on the sample. For example, according to [1] composites of immiscible metals began to use as contacts in vacuum switches since the 1960s. The classical methods of powder metallurgy do not allow introducing something new in the structure and properties of such materials. The SPS method owing to the ability of preserving the nanostructure in bulk material again raised interest in contact materials, allowing the creation of new materials with enhanced properties [2, 3]. However, a fundamental basis on the consolidation processes of mutually insoluble metals under SPS conditions is still not well undestood. The present work is devoted to the study of the structure formation processes of a Cu-Cr composite material under SPS conditions.

2. Methods

Copper (99.5 %, d < 100 μm) and chromium (99.1 %, d < 125 μm) powders were used as the initial materials. The composite Cu/Cr powders were obtained in the High-Energy Planetary Ball Mill "Activator-2S" (Activator, Novosibirsk, Russia). Premixed in a mortar Cu and Cr powders were subjected to the high-energy ball milling (HEBM) under an argon atmosphere in steel aqueous cooled jars, containing 7 mm steel balls. The ball-to-powder weight ratio was 20 : 1. Samples were treated in three cycles as follows: 20 min HEBM with subsequent cooling for 10 min at a total HEBM time of 60 min. The sun wheel rotation speed during the milling was 694 rpm at the jar-to-sun wheel speed ratio (K coefficient) equal to 1. Composite Cu/Cr powders were consolidated in a spark plasma sintering device (SPS, Labox 650, SinterLand, Japan). Treated powder placed in a cylindrical graphite die (inner diameter of 15.4 mm, outer diameter of 30 mm, and height of 30 mm). A 0.4-mm-thick graphite paper was placed between the sample and the die to form walls. The maximum load was 50 MPa. The d.c. (400 A) was fed through the sample and the die with the pulses of 40 μs; the intervals between pulses were 7 μs. The heating rate was 100°C/min, the sintering temperature was 700 °C, and the dwelling time was 10 min. During the SPS, the following experimental parameters were measured: temperature, pressure, current, voltage, displacement of the electrode, and vacuum values in the chamber. The thermocouple was inserted in a special hole in the graphite die (diameter of 2 mm, depth of 4 mm), which enabled to measure the temperature at the sample`s surface.

The structure of sintered materials were analyzed by means of scanning electron microscopy (SEM) on the JSM7600F (JEOL, Japan). The X-ray diffraction analysis was conducted on a Difray 401 diffractometer (Scientific instruments, Russia). The density of samples after sintering was evaluated via the hydrostatic weighing. The specific electric resistance was measured on a setup provided by KRIOTEL Ltd (Russia) by the four-contact method at the d.c. The hardness of the sintered samples was measured on the

polished samples via the Vickers method in accordance with GOST 2999-75 by means of a digital hardness meter HVS-50. A pressure of 5 kgf was applied to the sample surface for the specified time duration with a right quadrangle diamond pyramid.

3. Results and discussion

The effect of SPS parameters on the structure of materials obtained from composite particles of immiscible metals was investigated. It was shown that during the consolidation the structure of the particles changes: heating leads to the growth of chromium particles from 5-10 to 200-300 nm, also in the material appear copper inclusions along the boundaries of sintered powders. These inclusions can already be observed at 400 °C and their number increases with the temperature increase. At 700 °C its volume fraction reaches 10 – 12 % and then does not change with the increasing of the dwelling time. The formation of such inclusions can be explained by the popular theory that involves the occurrence of sparks between the particles of the sintered material under the action of current pulses. This leads to a strong local overheating and fusion of the particles.

Therefore, samples were also obtained by isolating the material in the matrix with alumina-silica fiber. During sintering according to this scheme at 700 °C shrinkage of the material continued for another 5 minutes of dwelling. Meanwhile in the case when the current was passed both through the matrix and through the sample at 700 °C shrinkage ceased during the first minute of dwelling, but it is worth noting that in this case shrinkage begins earlier that in the isolated sample. However, the structures of materials obtained by both schemes are the same. Despite the fact that in the second case sample was isolated and no current pulses were passed through it, copper inclusions along the grain boundaries are also found in its structure, which casts doubt on the assumption of sparks or breakdowns between particles in this material.

4. Conclusions

It is shown that SPS conditions make it possible to accelerate the material compaction process due to uniform heating throughout the sample volume. The formation of copper inclusions is caused by the desire of the system to reduce the number of interphase boundaries and to reduce other defects in the structure. Similar conclusions were reached in the work [4].

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Composite materials; immiscible metals.