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THE ROLE OF CHROMIUM MASS TRANSFER IN THE STRUCTURE FORMATION OF (Cu-Sn)-Cr-C MATERIALS DURING LIQUID-PHASE SINTERING

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Abstract

The effect of mass transfer of chromium on the structure formation of (Cu-Sn)-Cr-C materials in liquid phase sintering has been studied. For this, mixtures of powders of Cu90Sn10 tin bronze, commercially pure chromium, and graphite were compacted by static pressing. Some powder mixtures were mechanically activated in a ball centrifugal mill before compacting. The samples were sintered in vacuum at 720-920°C for 40-120 minutes. It has been found that the key role in formation of the structure of (Cu-Sn)-Cr-C materials in liquid phase

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sintering belongs to two processes: mass transfer of chromium via the liquid phase and the formation of chromium carbide. In sintering of mechanically activated (Cu-Sn)-Cr-C powder materials, these processes lead to dissolution of the initial deformed chromium particles and transfer of their substance to nanoparticles of graphite acting as crystallization centers for Cr_7C_3 chromium carbide. As a result, (Cu-Sn)-Cr-C materials have been obtained consisting of a bronze matrix and uniformly distributed in it Cr_7C_3 carbide particles, sized 50 to 700 nm.

Introduction

In mechanic engineering, diamond-abrasive tools with metallic matrices are used for machining ceramic blanks, as well as for shaping and dressing of grinding wheels [1, 2]. High hardness, wear resistance, and secure retention of diamonds are expected from matrices of these tools. So, carbide-forming metals, such as vanadium, niobium, titanium, and chromium, are added to matrices based on copper alloys, which improves adhesion of the matrix to diamond grains [3]. When manufacturing diamond tools by the powder metallurgy method, micro- and nanopowders of carbides of high-melting metals, cubic boron nitride, carbon nanotubes, and basalt fibers are introduced into the composition of metallic matrices to enhance their hardness and resistance to wear [4-7]. In works [8, 9], it is demonstrated that introduction of high-melting nanoparticles into metallic matrices of diamond-abrasive tools allows considerably enhancing wear resistance of the matrices and security of diamonds retention in them.

However, the use of nanopowders as modifying additives is associated with a number of difficulties. Their high chemical activity leads to the necessity of protecting the powders against oxidation during storage and use. Next, the tendency of nanopowders for clustering can hinder their mixing and uniform distribution in the batch mixture.

One of the solutions for this problem is in situ synthesis of reinforcing nanoparticles in the matrix of diamond and metal composite during sintering. For example, it is shown in [10] that introduction of carbon-containing additives into the Cu-Sn-Ti powder material allows obtaining titanium carbide nanoparticles during sintering which reinforce the matrix.

(Cu-Sn)-Cr-C powder materials are promising binders for diamond-abrasive tools. The basis of these binders is tin bronze with its good mechanical properties and thermal conductivity. Chromium combined with carbon is introduced to form solid carbides and improve retention of diamonds. Solubility of chromium in copper and its alloys in the solid condition is close to zero, which hinders obtaining sintered materials with a uniform structure [11]. This is why for the formation of chromium carbides evenly distributed within the matrix, stepped up mass transfer of chromium has to be provided during sintering.

The objective of this study is to find out the effect of mass transfer of chromium on formation of carbide nanoparticles during sintering of the (Cu-Sn)-Cr-C material.

Equipment, Materials and Research Methods

The following powders were used for preparing (Cu-Sn)-Cr-C powder mixtures: pulverized powder of Cu90Sn10 tin bronze (with the average particle size being 32 μm); commercially pure chromium powder with 10-150 μm sized splintery particles; graphite powder with 1-100 μm sized particles. The weight % proportion of mixture components was as follows: 90 - Cu90Sn10 bronze; 9 - Cr; 1 - graphite. In the double alloys diagram, this proportion of chromium and carbon corresponds to stable carbides Cr_7C_3 and Cr_3C_2 [12]. A part of the powder mixture was mechanically activated in the AGO-2U ball planetary mill for 300 minutes at the planetary mechanism rotation speed of 800 RPM. So, two batches of samples were prepared: one from the powders without mechanical activation, the other one from the mechanically activated powders. The samples were compacted by unilateral static pressing at the pressure of 570 MPa. The samples were sintered in vacuum at 720-920°C for 40-120 minutes. Then, microstructure of the samples and distribution of elements in them were studied using the EVO HD 15 scanning electron microscope (by Carl Zeiss) equipped with the energy dispersive X-ray microanalysis (EDXMA) system Inca Energy 350X-Max 80 (by Oxford Instruments). Phase composition of the sintered materials was explored by X-ray diffraction (XRD) analysis using the D8 Discover diffraction meter (by Bruker).

Results and Discussion

Phase diagrams of Cu-Sn-Cr and Cu-Sn-Cr-C multicomponent systems have not been discussed in literature yet [12], so the approximate idea about phase transformations in sintering of (Cu-Sn)-Cr-C materials can be gained from binary diagrams. For copper and tin alloy containing 10 weight % of tin, the solidus and liquidus temperatures are 835 and 1010°C, respectively [12]. This is why sintering of (Cu-Sn)-Cr-C materials proceeded in the solid phase at its temperature of 720°C, which is much lower than the solidus of bronze. XRD analysis of the sintered materials has returned the following phases: solid copper-based solution, metallic chromium, and graphite. No chromium carbides have been found.

Microstructure of the samples sintered at 720°C and 40 minutes exposure is shown in Figure 1. The shape and sizes of chromium particles have not changed in sintering. Chromium particles in the material sintered from powders without mechanical activation have splintery shape. Meanwhile, the material sintered from mechanically activated powders demonstrates smaller deformed particles of chromium. Both materials have no diffusion areas at the interface between chromium particles and bronze matrix. In the material sintered from mechanically activated powders, thin layers of graphite nanoparticles can be noted between particles of bronze.

Thus, no signs of dissolution of chromium particles and their chemical interaction with carbon are observed in the materials sintered at 720°C.

The 920°C sintering temperature falls within the solidus-liquidus range of Cu90Sn10 bronze. According to the Cu-Sn phase diagram for the said bronze, weight percentage of the liquid phase is around 45% at this temperature [12]. For chromium in the tin bronze at 920°C, the solubility value is unknown. In the liquid tin at 800-1000°C temperatures, significant solubility of chromium is observed, amounting to 1.7 to 6.5 at % [13].

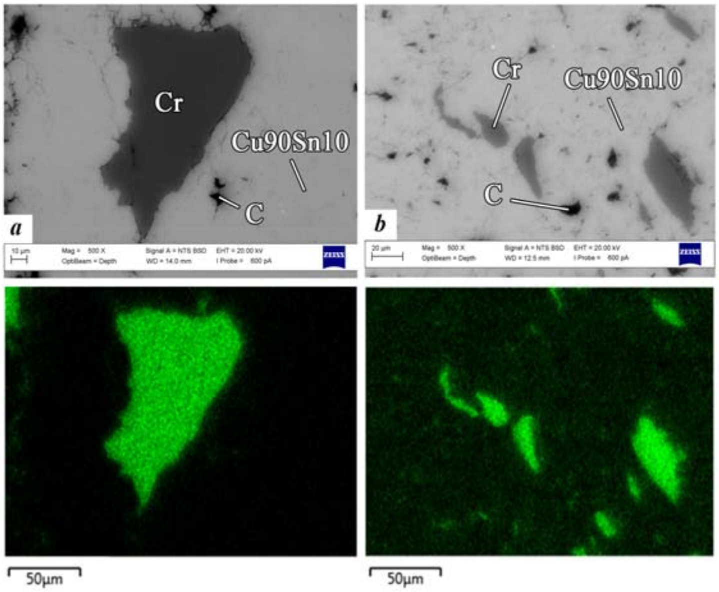

Figure 1. Microstructure of (Cu-Sn)-Cr-C materials sintered at 720°C: (a) powders without mechanical activation; (b) mechanically activated powders.

Figure 2 presents microstructure of the material sintered at 920°C from powders which were not mechanically activated. As it can be seen, a uniform chromium-containing coat was formed on the particles of graphite after the liquid phase sintering. The presence of this coat confirms the transfer of chromium via the liquid phase and its reprecipitation on the surface of graphite particles.

Mechanical activation of powders produces a noticeable effect on structure formation of (Cu-Sn)-Cr-C materials in liquid phase sintering (Figure 3). Instead the deformed chromium particles which can be seen in Figure 1, the material sintered at 920°C features groups of equiaxial particles sized 50 to 700 nm. If the duration of sintering is increased from 40 to 120 minutes, however, this does not have any essential effect on microstructure of the material.

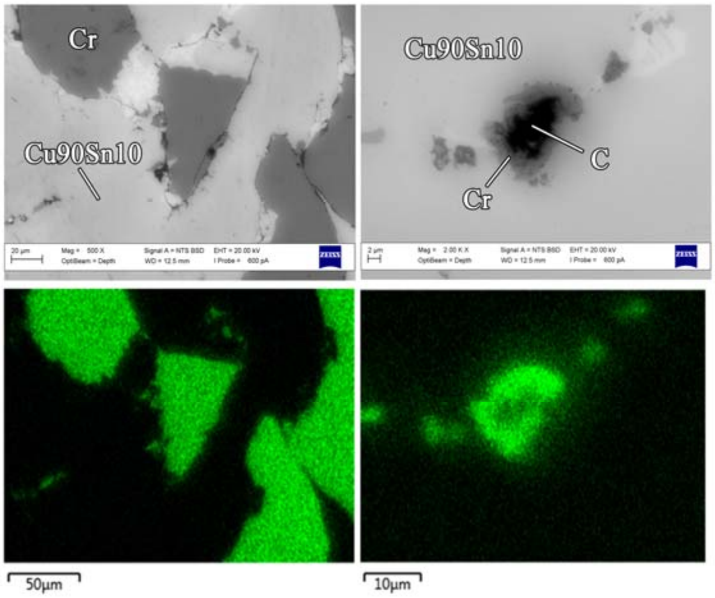

Figure 2. Microstructure of the (Cu-Sn)-Cr-C material sintered at 920°C, powders without mechanical activation.

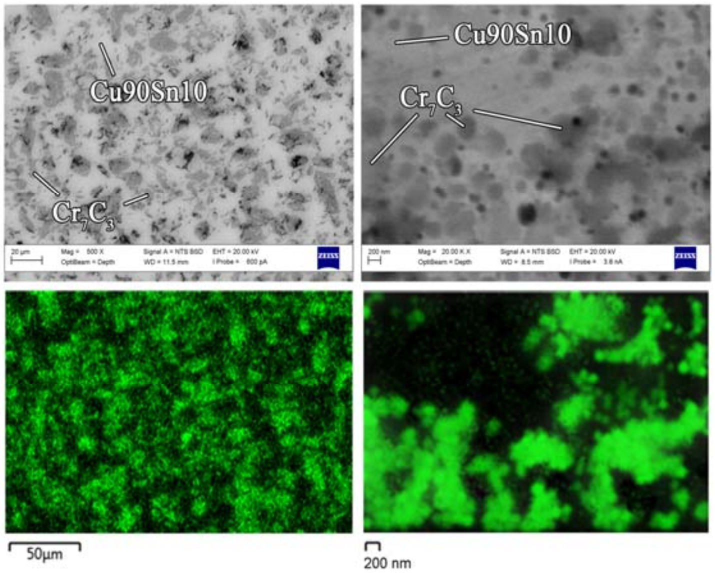

Figure 3. Microstructure of (Cu-Sn)-Cr-C material sintered at 920°C from mechanically activated powders.

Using XRD analysis, the authors have found that the samples subjected to 920°C liquid phase sintering consist of the following phases: solid copper-based solution, metallic chromium, and the Cr_7C_3 chromium carbide. A small quantity of the Cu_2O copper oxide is present in the samples, too. Figure 4 shows a detail of X-ray diffraction diagram of the sintered material in question.

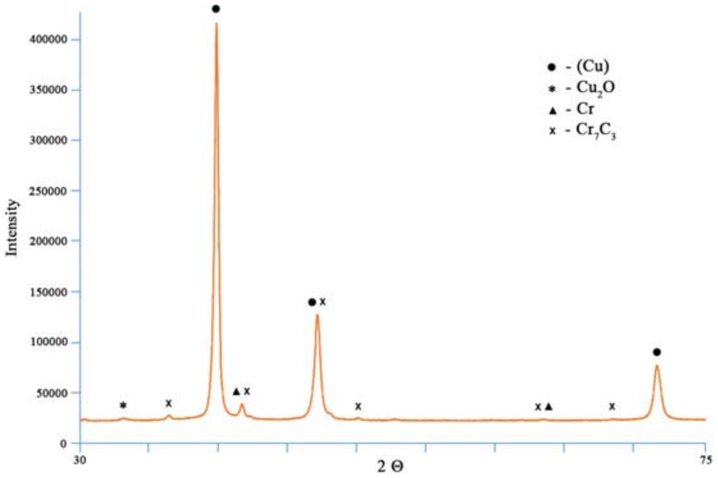

Figure 4. X-ray diffraction pattern of (Cu-Sn)-Cr-C material sintered at 920°C from mechanically activated powders.

EDXMA has demonstrated that finely dispersed particles which can be seen in Figure 3 contain 91-92 wt. % of Cr and 8-9 wt. % of C. This allows identifying them as particles of Cr_7C_3 chromium carbide.

Apparently, the chromium carbide particles have formed in the following way. At the temperature of 920°C, the bronze matrix of the (Cu-Sn)-Cr-C material partially melted. The liquid phase dissolved a certain quantity of chromium. Bronze melts are known to not wet graphite; however, if carbide forming metals are added to them, the contact angle of wetting is decreased [14]. The presence of dissolved chromium in the liquid phase contributed to wetting of graphite particles and carbides forming at the liquid phase to graphite interface. As a result of dissolution-reprecipitation of chromium, a chromium carbide coat which can be seen in Figure 2 is formed on graphite microparticles. In the material compacted from mechanically activated powders, graphite is available in the form of nanoparticles (see Figure 1). The free surface area of such particles is essentially larger as compared to graphite microparticles. Increase of the free surface area of graphite yields more extensive reprecipitation of chromium from the liquid phase. In its turn, chromium depletion of the liquid phase intensifies dissolution of chromium particles. The diagram of mass transfer of chromium during sintering of the (Cu-Sn)-Cr-C powder material is shown in Figure 5.

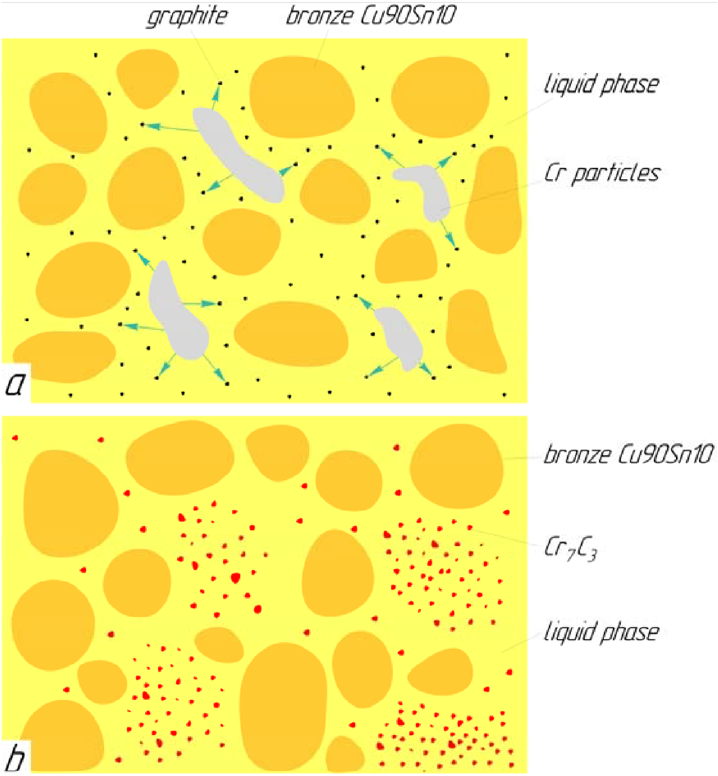

Figure 5. Scheme of structure formation of (Cu-Sn)-Cr-C materials during liquid-phase sintering: (a) mass transfer of chromium; (b) formation of Cr_7C_3 carbide.

Mass transfer via the liquid phase leads to dissolution of the initial deformed chromium particles observed in Figure 1 and formation of equiaxial carbide particles, with nanoparticles of graphite as crystallization centers.

Conclusion

Two processes, mass transfer of chromium via the liquid phase and chromium carbide formation, play the key part in formation of the structure of (Cu-Sn)-Cr-C materials in liquid phase sintering. In sintering of mechanically activated (Cu-Sn)-Cr-C powder materials, these processes lead to dissolution of the initial deformed chromium particles and transfer of their substance to nanoparticles of graphite acting as crystallization centers for Cr_7C_3 chromium carbide. As a result, (Cu-Sn)-Cr-C materials have been obtained consisting of a bronze matrix and uniformly distributed in it Cr_7C_3 carbide particles, sized 50 to 700 nm.

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