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Materials Science

Effect of Pressure on the Optimal Sintering Temperature under Hot Pressing Conditions of the Fe–Cu–Ni–Sn–Ti Alloy

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In the present work, the effect of P-T parameters on the physico-chemical and physico-mechanical characteristics of the sinters was studied during the hot pressing of Fe-Cu-Ni-Sn-Ti metallic composition. It was established that when the Ti content of the adhesively active elements to carbon is ≤15%, under the conditions of increased pressure (≥80 MPa), it was possible to form samples with high characteristics (density, hardness, microhardness) during solid state sintering. It is shown that by increasing the pressure to 45 MPa or up to 120 MPa, it is possible to fabricate a sintered composition with maximum indicators at a significantly lower temperature (700°C). *© 2024 Bull. Georg. Natl. Acad. Sci.*

pressure, temperature, hot pressing sintering, adhesion

Currently, in a number of industries (construction, geology, etc.), one of the most widely used classes of composites is diamond composite materials (DCM). The quality and performance properties of DCM depend both on the consistency of the diamond+metal composition (binder) and on the technology for its production. As is known [1,2], a binder must satisfy a number of technical requirements.

One of the most important criteria when selecting the composition of the binder is that in the stage of fabrication, the temperature of hot pressing or sintering of the binder should be minimal and, taking into account the heat resistance of diamonds, not exceed 800-850°C.

The diamond retention strength is one of the most important conditions for the performance of DCM. As is known, diamond crystals are held by the matrix, firstly, due to the mechanical enfolding by the bond material and secondly, by the adhesive forces of the metal-diamond contact surface.

The mechanical retention of diamonds is largely determined by the strength of the enfolding of diamond grains with the metal binder and depends on the mechanical and thermophysical (strength, hardness, coefficient of linear expansion, heat resistance) properties of the metal or alloy [3].

The use of adhesive materials in relation to diamond (carbon) at the same time as high-temperature metals such as Ti, Cr, Zn and W, etc. in order to increase the holding strength of diamonds, as well as to increase the physical and mechanical properties of the metal binder is associated with the need to increase the sintering temperature of DCM. This factor limits their percentage in DCM binders to a minimum level. Usually applied metal compositions such as copper, tin bronze, copper-nickel, and Ferro-copper alloys, have very low adhesion to diamond and, therefore, a weak bond between the diamond grain and the binder.

On the other hand, it is known that even minor additions of adhesive-active elements to the composition of inactive alloys lead to a sharp decrease in the contact angle of diamond surfaces with the melts of these alloys and thereby increase the adhesion forces. Thus, the presence of even 0.5% Ti or Cr in the Cu-Sn alloy (ϴwt.≈130°), which is inactive with respect to diamond (graphite), leads to a sharp improvement in the wetting of diamond surfaces (ϴwt.≈0°) and adhesion forces. But in such compositions containing active metals such as Ti, Cr, Zn and W, due to their low content, do not have the desired noticeable effect on the physical and mechanical properties of the binder, therefore they have low hardness and, accordingly, wear resistance, which limits the scope of their application [4].

The production of DCM of the desired composition with specified physico-mechanical and physico-chemical properties, under conditions of limited sintering temperature, is possible only after introducing new methods or certain innovations in the technology of their fabrication (i.e., after solving a number of technical and technological problems).

Currently, hot pressing is one of the main methods for producing DCM with a “refractory” metal binder. Although molds for hot pressing are made from special, high-strength graphite, the operating pressure during sintering usually does not exceed 400-700 kg/cm^2. Heating is carried out by directly passing current either through the punches and the sample, either through the mold matrix, or by induction currents.

The equation for quantitatively assessing a material densification rate during hot pressing has the form [5,6]:

$$\frac{d\mu}{dt_{hp.}} = \frac{d\mu}{dt_{p+s}} + \frac{3p}{4\eta}(1-\mu),$$

where $(d\mu/dt)_{hp.}$ is the rate of compaction during hot pressing, $(d\mu/dt)_{p+s}$ is the rate of compaction of the substance during conventional sintering, μ is the relative density, p is hot pressing pressure, η is material viscosity at process temperature.

From the expression, it follows that pressure is one of the main factors influencing the hot pressing process flow.

Work [7] shows the determining role of hot pressing pressure on the degree of compaction during solid-phase sintering of Inconel 718 super alloy (Ni-Cr-Fe base) in the pressure range 50 MPa-110 MPa and temperatures 950-1200°C. It is shown that at moderate temperatures 950°C the main driving of densification is plastic flow. When temperature rises at that stage, dominated dislocation creep and diffusion creep occur. It has been established that for a given alloy composition, maximum properties are realized not at maximum sintering parameters (P = 110 MPa and T = 1250°C), but lower, when P = 70 MPa and T = 1150°C.

When obtaining a composition containing diamond crystals, as is known, there is a limitation on the pressure (hot-pressing tooling) and temperature (degradation and graphitization of diamond) of the process.

Then, it is very important to identify the influence of pressure on the sinterability of the metal composition at relatively low temperatures (≤800°C) that do not cause degradation of diamonds (loss of strength) and the performance of hot pressing equipment (molds).

The experiments were carried out on a hot pressing unit developed at the GTU with electrical heating, power 25 kW and 30-ton force. The developed mold design made of special heat-resistant steel with graphite-ceramic elements. It made possible to increase the range of applied pressure at high temperatures (800-870°C) up to 120 MPa [8].

The influence of pressure on the properties of the Fe-Cu-Sn-Ti alloy sintered at a temperature T=770°C, with a Ti content of about 15 wt. %, was studied. Ti is chemically active towards carbon (diamond), thereby imparting adhesive activity to the alloy in relation to the surface of diamond crystals.

The choice of titanium to impart adhesive properties to the metal bond was justified by the following factors:

1. Ti actively reacts with carbon (diamond) at relatively low temperatures, thereby forming a transition carbide layer between the diamond and the binder, increasing the adhesion forces of the crystals to the matrix [9].

2. The addition of titanium leads to the formation of intermetallic phases (with Cu, Fe) which increases material hardness and as a result wear resistance.

During the sintering process, titanium can react either with the diamond carbon or binder components (primarily with iron). Under hot pressing conditions, taking into account the thermodynamic potentials of the formation of titanium carbide (-44 kJ/mol) and FeTi intermetallic compound (-170 kJ/mol), the interaction of Ti with the diamond surface is more likely.

For the Fe-Cu-Sn-Ti system with Ti content of 15%, the liquid phase is fixed no earlier than 860-870°C. Conducting experiments at 770°C guaranteed solid-phase sintering of the samples. The specificity of solid-phase sintering is that the adhesive activity of the metals present in the system in this case manifests itself in direct contact with the surface of the diamond (graphite). In this case, a chemical bond is formed with the formation of a strong transition zone (carbide). The adhesion strength to the diamond in this case will depend on the size of the contact surface (volume fraction of the active element) and the tightness of the Me – C surfaces (on the degree of compaction of the bond) and depends on the P – T parameters of the sintering (process).

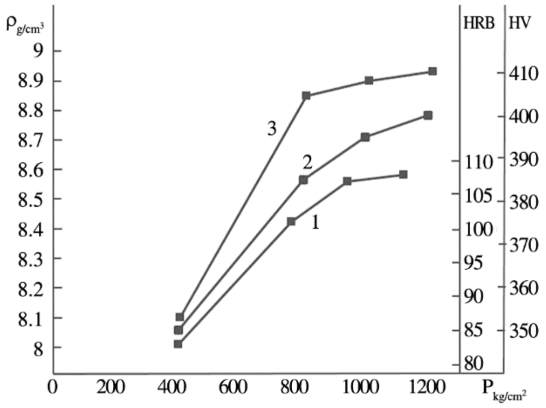

Fig. 1. Change in density ρ (1), hardness HRA (2), and micro hardness HV (3) of sintered samples of the Fe–Cu–Ni–Sn–Ti system depending on the pressure (P) of hot pressing at T=770°C.

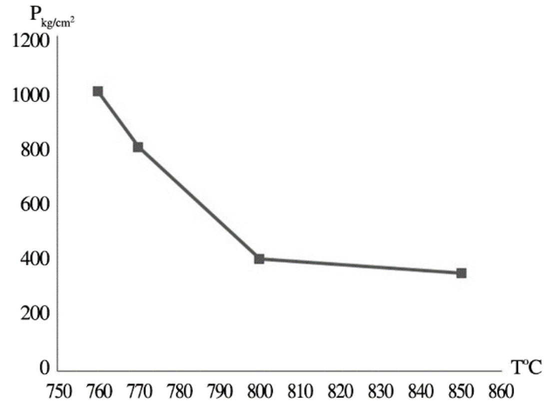

Fig. 2. Changing the required sintering temperature from pressure to achieve maximum values of ρ, HRA, and HV for the Fe – Cu – Ni – Sn – Ti system.

The data show that under solid-phase sintering conditions, pressure is one of the determining factors influencing the process of alloy structure formation (Fig. 1). An increase in pressure from 400 kg/cm^2 to 800 kg/cm^2 has a particularly significant effect on such physical characteristics of sintered samples as hardness and density. This indicates a sharp intensification of the sintering process of the metal composition at a constant temperature. A further increase in pressure to 1000 kg/cm^2 does not lead to significant changes in the properties of the samples, which indicates that a structure with maximum properties has been already obtained (for a given alloy composition).

Similar studies carried out at temperatures of 790, 820 and 850°C in the range of 350-1000 kg/cm^2 during sintering of the Fe-Cu-Sn-Ti composition showed that the maximum possible values of ρ, HRA, and HV with an increase in hot pressing pressure, can be also realized at lower temperatures (Fig. 2).

The obtained data showed that during solid-phase sintering of DCM under hot pressing conditions, the pressure factor is one of the determining factors and opens up prospects for the fabrication of diamond-containing composites with adhesive active binders having high physico-chemical and mechanical properties.

მასალათმცოდნეობა

წნევის ზეგავლენა Fe – Cu – Ni – Sn – Ti შენადნობის ცხელი დაწნეხვით შეცხობის ოპტიმალურ ტემპერატურაზე

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წინამდებარე ნაშრომში შესწავლილია Fe – Cu – Ni – Sn – Ti ლითონური კომპოზიციის ცხელი დაწნეხვით შეცხობის პროცესზე P–T პარამეტრების გავლენა ცხობილის ფიზიკურ-ქიმიურ და ფიზიკურ-მექანიკურ მახასიათებლებზე. დადგენილია, რომ ნახშირბადის მიმართ ადჰეზიურად აქტიური ელემენტების Ti-ის შემცველობისას ≤15% გაზრდილი წნევების პირობებში (≥80 მპა) შესაძლებელია მყარფაზა შეცხობისას მაღალი მახასიათებლების (სიმკვრივე, სისალე, მიკროსისალე) ნიმუშების ფორმირება. ნაჩვენებია, რომ წნევის მატებით 45 მპა ან 120 მპა-მდე შესაძლებელია მაქსიმალური მაჩვენებლების მქონე შემცხვარი კომპოზიციის მიღება მნიშვნელოვნად (700°C) უფრო დაბალ ტემპერატურაზე.

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