Diamond and Low Carbon Steel Interaction at Melting Temperature of Fe-C Eutectic

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Abstract. The structure, elemental and phase composition of the products formed during the contact interaction of diamond with low-carbon steel in vacuum at the Fe-C eutectic melting temperature were studied. Cylindrical tablets made of low carbon steel (less than 0.1 % wt. C), and pyramid shaped natural diamond crystals were used as a contact pairs. Diamond crystals were mounted by their flat base on the horizontal surface of steel tablet and a load was applied to the diamond crystals top. Contact samples were sintered in a vacuum furnace at a maximum heating temperature of ~1165 °C. Sintered diamond-steel tablet samples were studied by optical and scanning electron microscopy, X-ray diffraction analysis. It was shown that the initial steel tablet with a ferrite-perlite structure passed cementation during sintering in contact with diamond. The most intensive cementation is undergone by the non-melted upper layer of the steel tablet ~110 μm thick, which adjoined the Fe-C eutectic melt during sintering. The microhardness of this layer was ~4945 MPa. As it deepens into steel tablet a gradual transition of structure from perlite-cementite to perlite and further to the initial ferrite-perlite microstructure is observed. Herewith, the microhardness changes from ~ 4945 to 1570 MPa.

1. Introduction
One of the types of contact interaction of diamond with metals of the 8th group, which ensures a strong adhesion of diamond with the tool matrix during sintering, is the phenomenon of eutectic melting at the diamond-metal interface [1, 2]. For the first time, the fact of the formation of a liquid phase during high-temperature contact of diamond with metals of the iron triad, which in addition to iron includes cobalt and nickel, was experimentally demonstrated in the work [1]. In [1, 2] it is shown that the temperature at which the liquid phase formation begins at the diamond-metal interface is in accordance with the melting temperatures of the corresponding metal-carbon eutectics (Me-C). As is known, the melting points of the eutectic Co-C and Ni-C alloys significantly exceed the threshold temperature at which even natural diamonds with high heat resistance (compared with synthetic ones) lose their strength. The lowest melting point of the eutectic from the iron triad group is in the Fe-C system, which is only 1153 °C, which is lower than the heat resistance of even some types of synthetic diamonds [1–5]. In this regard, and also taking into account the problem of the strong fixing of diamond single crystals in iron-based matrices [6, 7], it is interesting to consider and study the contact
interaction of diamond with low-carbon steel when heated in vacuum to the Fe-C eutectic melting temperature.

The purpose of this work is to study the structure, elemental and phase composition of products formed during the contact interaction of diamond with low-carbon steel in vacuum at the eutectic Fe-C melting point.

2. Experimental procedures
Cylindrical tablets with a diameter of 8 mm and a height of 9 mm made of low carbon steel were used to study the contact interaction of diamond with iron during heating in vacuum. Pyramid-shaped diamond crystals were used as contact pairs for steel tablets, selected from waste of lapidary production (industrial waste - crystals unsuitable for cutting into brilliant). Diamond crystals with their flat bases were mounted on flat horizontal surfaces of steel tablets. Herewith, a load was applied to the diamond crystals top. The load value for each pair of diamond-steel tablets was chosen so that the pressure exerted by diamond crystals having different sizes, respectively, different base areas, was ~117 kPa. Samples in this position were placed in a vacuum furnace (SNVE-1.3.1/16I4, MEVZ, Russia) and were sintered. Figure 1 (a, b) schematically shows the relative position of the diamond crystal and the steel tablet before and after sintering. A vacuum furnace provided a residual pressure of ~6.4 mPa at room temperature. At the maximum heating temperature, the residual pressure in the furnace chamber was ~72 mPa. At the initial stage, the diamond-steel tablet contact pairs were heated up to 600 °C with a rate of 10 °C/min and followed holding at this temperature for up to 30 minutes. Then the temperature was increased with the same rate to 900 °C, upon reaching which holding was carried out for 30 minutes. Heating to a maximum temperature of ~1165 °C was carried out with a rate of 5 °C/min. After holding for 5 minutes at 1165 °C, the furnace was turned off, and the chamber temperature decreased in free cooling mode.

![Diagram](image)

**Figure 1.** Scheme of diamond crystal and a steel tablet position in a vacuum furnace before (a) and after sintering (b): 1 – a diamond crystal, 2 – a steel tablet, 3 – a solidified Fe-C eutectic alloy.

The structure of the steel tablet, the Fe-C eutectic alloy formed at the diamond-metal contact zone under given conditions, and the diamond-metal interface were studied by optical and scanning electron microscopy (SEM) using Neophot-32 (Carl Zeiss, Germany) and TM3030 devices (Hitachi, Japan). The chemical composition of the initial steel tablets was determined using a Foundry-Master UVR optical emission spectrometer (WAS AG, Germany). X-ray diffraction analysis of steel tablets before and after interaction with diamond at high temperature was performed on a D8 Discover diffractometer (Bruker, Germany) using CuKα radiation (λ=1.541 Å). The diffraction spectra were recorded in the range of angles 2θ 10°–90º with step size 0.02°. The diffraction spectra were processed in the Crystallographica Search-Match (CSM) program using the Crystallography Open Database (COD).

The microhardness of the steel tablets samples before and after contact interaction with diamond at high temperature was measured by the Vickers method on a PMT-3 (LOMO, Russia) at a load of 0.49 N. Microhardness values were averaged over at least 10 values.
3. Results

3.1. The composition and structure of the initial steel tablet

Table 1 presents the results of a chemical analysis of the initial steel tablet used in the experiments. As follows from the table the chemical composition of tablet corresponds to steel grade st1sp according to GOST 380-2005 with a carbon content of 0.09 wt. %, commonly used in the manufacture of nails, wires, rivets, etc. [8, 9].

| The content of chemical elements, wt. % |
|----------------------------------------|
| C    | Si    | Mn    | Cr    | Ni | P   | S  | Cu | Fe  |
| 0.09 | 0.25  | 0.40  | 0.02  | 0.02 | --  | -- | 0.03 | rest |

Figure 2 shows SEM images of the initial steel tablet (before sintering with diamond) structure (a, b) and X-ray diffraction pattern (c). It is seen that the initial metal tablet has a typical low carbon steels structure, consisting of grains of ferrite and perlite. Ferrite grains have a quasi-axial shape with an average size of ~15 μm. The volume fraction of perlite does not exceed 15 %. Perlite is mainly located along the boundaries of ferrite grains and has a plated structure. The average distance between the plates lies in the range of 0.8–1.0 μm. The microhardness of the initial steel tablet, averaged over 20 points, is 1570 ± 40 MPa.

The diffraction pattern contains three intense peaks at 2θ angles ~44.69º, 65.05º and 82.37º reflected from the (110), (200) and (211) planes of the BCC lattice of Fe. Six weak peaks at the noise level in the 2θ angle range ~37.78º–49.11º indicate the presence of an insignificant amount of the carbide phase – cementite (Fe3C), which is the structural component of the perlite phase. A clear reflex at an 2θ angle of ~24.94º were identified as a graphite peak reflected from the (0001) plane.

![SEM images and diffraction pattern](image)

**Figure 2.** The structure of the initial steel tablet obtained on SEM at different magnifications (a, b) and its diffraction pattern (c): 1 – grains of ferrite, 2 – grains of perlite.
3.2. The appearance of contact samples after sintering

Figure 3 shows photographs of the appearance of diamond-steel tablet contact pair samples, taken from above and side. It is seen that a liquid phase of an iron and carbon eutectic alloy forms on the diamond-steel tablet interface during the heating process. Most part of the eutectic melt is squeezed out from under diamond crystal base under the action of applied to its top load, which leads to slight displacement down of diamond. The part of the eutectic melt remaining under the diamond crystal in the form of a thin layer, welds the diamond base to a steel tablet upon cooling. The extruded part of the eutectic melt, when solidifying in the form of a flattened drop, also welds a diamond crystal to the surface of a steel tablet.

The shear mechanical tests were carried out according to the procedure described in [10] to assess the contact strength of a diamond crystal with a steel tablet. However, it was not possible to directly determine the strength of the interfacial contact between diamond and steel tablet during tests, since, the fractures did not occur by the interphase contact, but by the diamond crystals in all the samples, which indicates the strong adhesion of diamonds to the steel tablet through a thin layer of solidified Fe-C eutectic.

![Figure 3. Appearance of diamond-steel tablet samples after their sintering in a vacuum furnace (view from above and side): 1 – a diamond crystal, 2 – a steel tablet, 3 – a solidified Fe-C eutectic alloy.]

3.3. The structure of the diamond-low carbon steel interphase zone

Figure 4 shows a photograph of a longitudinal section of a sintered diamond-steel tablet contact pair (a), microstructure images of steel tablet various zones (b–e) subjected to cementation during sintering and diffraction pattern obtained near the diamond-tablet contact zone (f). The longitudinal section (Fig. 4 a) of the sintered diamond-steel tablet is conventionally divided into layers by the microstructure character, which are indicated by horizontal dashed lines.

Figure 4 (b) shows the microstructure image in the zone of direct contact of the Fe-C eutectic with the diamond crystal and steel tablet. As can be seen the Fe-C eutectic by its microstructure is cast iron with unevenly distributed graphite mainly with plated form [11–14]. It is also seen that this layer is tightly adhered to the diamond crystal. The average microhardness is ~1706 MPa due to the relatively high content of structurally free graphite. The average thickness of the Fe-C eutectic layer was ~250 μm.

The steel tablet layer directly adjacent to the Fe-C eutectic layer, which did not melt during heating, underwent the most intensive cementation (Fig. 4 b) as a result of a significant acceleration of the diffusion of carbon atoms from the Fe-C eutectic melt [2, 9]. The microstructure of the cemented layer consists of perlite and cementite [15]. Cementite is emitted in the form of a fine grid along the boundaries of perlite grains. The average thickness and microhardness of this layer are, respectively, ~110 μm and ~4945 MPa. The diffraction pattern of this layer (Fig. 4f) contains three peaks at angles of 20 ~44.69°, 65.05° and 82.37°, corresponding to the planes (110), (200) and (211) of the α-Fe (BCC) lattice. Peaks at 2θ ~37.58°, 40.69°, 42.92°, 44.83° and 49.11° reflected from the planes (121), (201), (211), (031) and (122) indicate the presence of cementite (Fe3C).

Then a layer (Fig. 4 c), the microstructure of which consists of granular perlite follows. The average thickness of the perlite microstructure layer is ~350 μm, and its microhardness is 3543 MPa. The perlite layer is followed by an intermediate layer with a ferrite-perlite microstructure (Fig. 4 d), in which the amount of perlite gradually decreases as the depth into the steel tablet, while the number of
ferrite grains increases. Herewith, the microhardness decreases from 1943 to 1570 MPa with a decrease in the amount of perlite, that is the average microhardness of the initial steel tablet with a ferrite-pearlite microstructure (Fig. 4 e).

4. Conclusion
The structure, elemental and phase composition of the Fe-C eutectic formed in the process of high-temperature contact interaction of diamond with low-carbon steel in vacuum are studied. It was established that in the diamond-steel tablet contact zone a Fe-C eutectic melt is formed, a thin layer of which, when solidified, welds the diamond crystal to the steel tablet at the temperature-time mode specified in the experiment. It was shown that the initial tablet of low-carbon steel with a ferrite-pearlite structure is cemented during sintering in contact with diamond. The non-melted top layer of a steel tablet ~110 μm thick adjacent to the Fe-C eutectic underwent the most intensive cementation during sintering. The microhardness of this layer is ~4945 MPa. As it deepens into steel tablet a gradual transition of structure from perlite-cementite to perlite and further to the initial ferrite-perlite microstructure is observed. Herewith, the microhardness changes from ~4945 to 1570 MPa.

Figure 4. A photograph of a longitudinal section of a sintered diamond-steel tablet contact pair (a), microstructure images of steel tablet various zones (b–e) and diffraction pattern of the layer that undergone the most intense cementation (f).
5. References

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