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Original Article

Contact interaction of metal melts with fullerite and graphite



R.M. Nikonova, V.V. Lad'yanov

Udmurt Federal Research Center of the Ural Branch of the Russian Academy of Sciences, 34 T. Baramzina Str., Izhevsk, 426067, Russia

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ABSTRACT

Comparative studies of contact interactions of Fe, Ni, Cu melts with graphite and fullerite were performed using a sessile drop method, X-ray analysis and metallographic analysis. It was shown that the contact interaction of Fe with fullerite and graphite substrates caused changes in the structure of iron. Namely, perlite, cementite and graphite precipitates in different proportions were formed. In comparison with Fe, in case of Ni total soaking of the fullerite substrate by the metal (and only partial in case of graphite) was observed while metal structure remained unchanged. No contact interaction of Cu with fullerite and graphite occurred. The differences in wetting of carbon materials by metal melts were stated to be caused by the structural changes in fullerite at heating higher than 1000°C (its amorphization) and further increase of carbon substrate porosity. A scheme was presented illustrating the difference in contact interactions of metal melts with fullerite and graphite.

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1. Introduction

Much attention is currently given to the development of prospective technologies aimed at obtaining new carbon-containing composite materials with improved mechanical and thermo-electrophysical characteristics, namely those with matrices from carbon-reinforced metal alloys [1–6]. In process of their metallurgical production wetting at contact interaction of the metal base melt with carbon material is considered especially important.

Quite a number of publications have presented a detailed study of the peculiarities of physicochemical interaction of graphite and diamond with melts of metals and alloys [7–10]. As a rule, wetting of graphite and diamond by metal melts, such as Fe, Al, Ti, Nb, etc. occurs if metal fairly intensively

reacts chemically with carbon with the formation of carbides. Work of adhesion for different metals is well known and makes up 1.52 J/m² for Al-graphite at 1200°C, Fe-graphite –3.34 J/m², and Ni-graphite –3.9 J/m² at 1550°C [11]. There are some papers that are devoted to the investigation of wetting of graphite by liquid Al [12,13] and its alloys [14], and to the influence of a graphite coating on an alumina substrate [15].

Cu, Ag, Au, etc. are inactive to carbon, with work of adhesion of their melts being close to the energy of their dispersion interaction and no more than 0.2–0.3 J/m². Much attention is given in literature to the analysis of wetting of graphite by copper [10,16–18], also, in conditions of metal melting by ion beam [10], and glass carbon by Cu-Si melts [2]. To increase carbon dissolution in inert copper a third component (Ni, Co, Mo, etc.) is introduced into a metal. When, for example, Cr, Ti [18] and V [17] are introduced into liquid copper during wetting of the substrates of graphite and glass carbon, carbide phases

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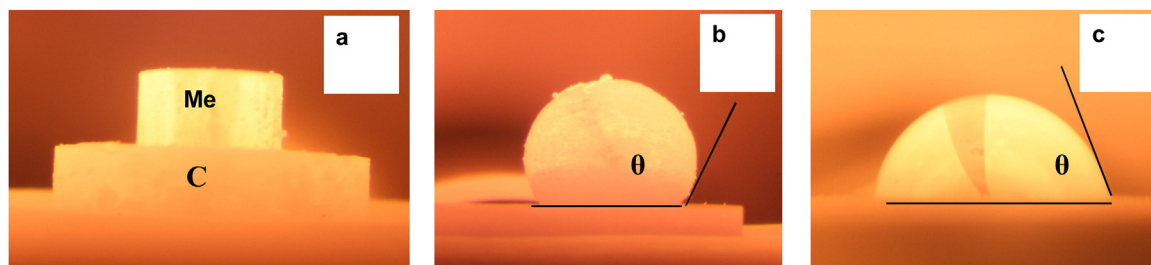


Fig. 1 – Sample of Me on carbon substrate before (a) and after metal melting (b,c).

are formed in a thin surface layer at the Cu-carbon interface. Composite materials where metal in normal conditions is non wettable and resistant to carbon penetration are produced by means of permeation of carbon at high temperatures and under excessive pressure in papers [3,4].

New research trends are now in focus. One of the methods to obtain alloys from immiscible components is contact doping during wetting [19–21]. For example, by doping Fe-Cu alloys with graphite during wetting it is possible to obtain multilayer composites with different structures. By varying cooling speed it is also possible to change the kinetics of a layering process to form a layered composite with a pre-eutectic and eutectic composition [21].

Paper [22] focuses on nucleation processes and diamond growth at the Ni-Mn - graphite interface.

A growth of publications has been observed lately on the wetting of other carbon forms by metal melts [23–29]. For example, the wetting of carbon nanotubes by iron [23], Co, Ni [24], and Hg [25]. There are some studies on the wetting of vitreous carbon by Cu-Si alloys [2], liquid Si-Ti [30] and Si-Co [31], pyrocarbon and pyrographite by Al-Si [32] and Si-Co alloys [31]. One of the papers described wetting of amorphous carbon films [5].

Fullerites, whose structure essentially differs from other carbon materials, are considered prospective nanoobjects for the production of new materials [33,34]. The potential application of fullerites in metallurgy requires the consideration of fullerites wettability by liquid metals and alloys. At the same time there are practically no fundamental studies on wetting-determined contact interactions of metal melts with fullerites.

This work presents a comparative study of the contact interaction of iron, nickel, and copper melts with carbon materials – graphite and fullerite. Special attention is given to the analysis of the stability of Me-C phase components. The analysis takes into account possible structural changes during heating both in metal melts and carbon materials used.

Besides, one of the goals of the present paper is working out of the procedure which will allow to reveal the differences in metal behavior on a graphite substrate taking into account big differences in saturated vapor pressure in case of graphite and fullerite.

2. Materials and methods

The investigation concerns metals having different melting temperatures and affinity to carbon - Fe, Ni, and Cu. Namely, refined iron ZhR 6 (ЖР6), high-purity Ni and Cu were used

(total impurity content ≤ 0.26 , 0.20 , and 0.015 %wt, respectively). The initial sample represented a $\varnothing 10$ -12 mm disk. Metal sample weight was determined by reference to carbon substrate sizes and was 0.30 – 0.35 g.

Graphite C_g and fullerite $C_{60/70}$ were taken as substrates. Graphite substrates were prepared from $\varnothing 10$ A.C.S. graphite rods so that both surfaces were plane-parallel. Fullerite substrates were prepared from fullerite powders with a laboratory press (under 5 MPa). Final, disk-shaped, substrates were ~ 5 mm high. An initial substance for the fullerite substrate represented a mixture of fullerites $C_{60/70}$. According to the high-performance liquid chromatography (HPLC) data the composition of the initial mixture $C_{60/70}$ was as follows: 82.18% - C_{60} , 14.08% - C_{70} , 2.81% - $C_{60}O$, $C_{60}O_2$ and $C_{70}O$; 0.93 Wt% - higher fullerenes C_{76} , C_{78} , C_{82} , C_{84} . According to the thermogravimetric analysis (TGA) the residual toluene made up 1.1 Wt% (as a crystalline solvate $C_{60}-C_{70}-C_6H_5CH_3$). Fullerites $C_{60/70}$ were prepared by electric-arc evaporation of graphite rods and subsequent extraction of fullerenes from fullerene-containing soot (standard Kretschmer-Huffman method [35]). The compared carbon substrates were slightly different in density ($\rho_{gr.} = 1.59$ g/cm³ and $\rho_{full} = 1.53$ g/cm³).

Surface topography studies of the carbon substrates used were carried out on a SOLVER P47 PRO atomic force microscope in contact mode. The arithmetic mean roughness (R_a) of the surface was calculated from images of 12 sections with a base size of 2×2 μ m for each sample using the software Image Analysis 3.5.0.

To study the metal-substrate contact interaction a sessile drop method was used in conditions of a simultaneous heating of the metal and the substrate. A general view of the metal sample on the substrate is presented in Fig. 1: a) before melting b), c) after melting till a drop is formed. Contact angle θ was chosen as the parameter of the contact interaction between the metal melt and the substrate material. The contact angle values can range from obtuse (b) to acute angles (c).

In order to reduce the influence of the magnetic field from a molybdenum heater, the samples were initially heated under vacuum ($P = 10$ -3 Pa). Heating under vacuum conditions led to complete destruction of a fullerite substrate (in the case of graphite it was not observed). The breakdown of the substrate at the investigated temperatures was caused by considerable fullerite sublimation. Taking into account a 26 orders difference between the values of saturated vapour pressure of fullerite and graphite at $T = 1200^\circ\text{C}$ [36], the samples were heated in a helium atmosphere (grade A - 99,995%) under excessive pressure P (He) in the range of 0.1 – 0.9 atm. The ful-

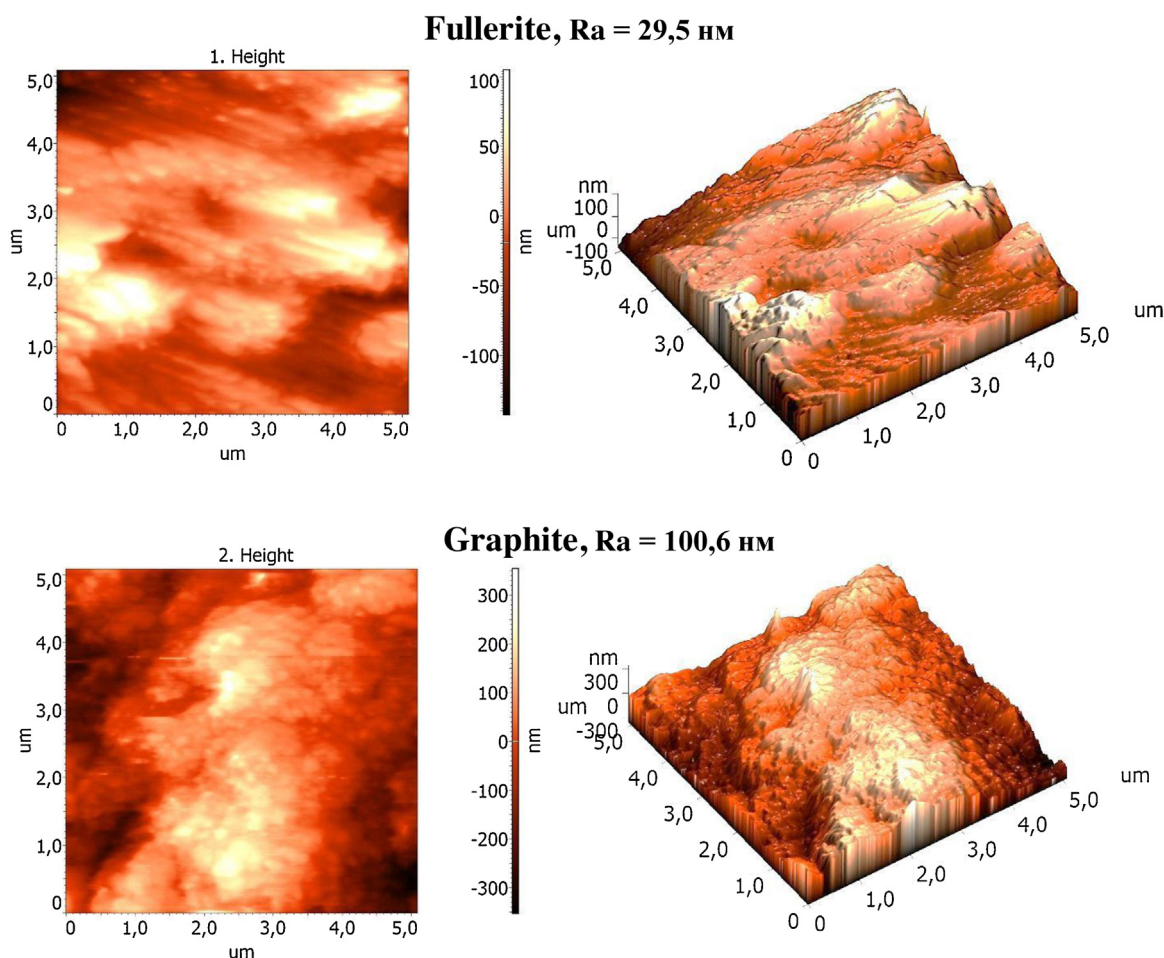


Fig. 2 – The roughness of the fullerite and graphite substrates in the initial state

literate substrate was found to remain unchanged only if the pressure P (He) was not less than 0.4 atm. Thus, all successive samples were heated in a purified helium atmosphere under excessive pressure ~ 0.4 atm. Helium passed through the system of two purifying furnaces (the first - with copper shavings, the second - with titanium sponge). Such a purification system guarantees a high inert gas purity.

For the system to achieve equilibrium, contact angles were measured during successive stepwise heating of the samples from 1000 °C to 1500 °C with a 50 °C interval and a 20 m exposure at each temperature. The maximum heating temperatures made up $T_{max} = 1500$ °C for Fe and Ni, and 1300 °C for Cu. Monitoring of the melting process was performed by means of an optical system built into the vacuum camera working in the regime of automatic shooting. Additional illumination of the drop, which is used in a number of works (for example [37]), was not applied. The drop was naturally illuminated at temperatures above 800 °C.

The interaction between the metal and the carbon substrate was monitored by the changes of contact angle on the surface of the carbon substrate, and also by the results of X-ray and metallographic analyses. Contact angles were determined by processing sample pictures; measurement error was 1.2%. X-ray studies were performed with a DRON-6 diffractometer (monochrome Cu-K α radiation). For metallographic analysis

the metal samples before and after the contact interaction with the carbon substrate were encapsulated into epoxy resin in a cell. An etching reagent for Fe and Ni was a 4% alcohol solution of nitric acid. An optical microscope NEOPHOT-21 was used to study the structure.

3. Results and Discussion

The results of the analysis of the roughness of the compared substrates in the initial state are shown in Fig. 2. It can be seen that the arithmetic mean deviation of the Ra profile of the surface of the fullerite substrate is about 3 times less than that of the graphite substrate and is 29.5 and 100.6 nm, respectively.

Comparative analysis of the wetting of different carbon substrates by metal melts demonstrated that different carbon modifications were wetted differently. Fig. 3 presents a general view of the samples after the experiment at $T = 1500$ °C for Fe and Ni, and 1300 °C for Cu. We can see distinctively different drop shapes for the system Me-graphite and Me-fullerite for the metals under study. In case of Fe there is only an insignificant difference in drop shapes after melting the metal on the fullerite and graphite substrates. In case of Ni the melt totally penetrated into the fullerite substrate, while graphite penetrated only partially. In contrast with Fe and Ni, the shape and

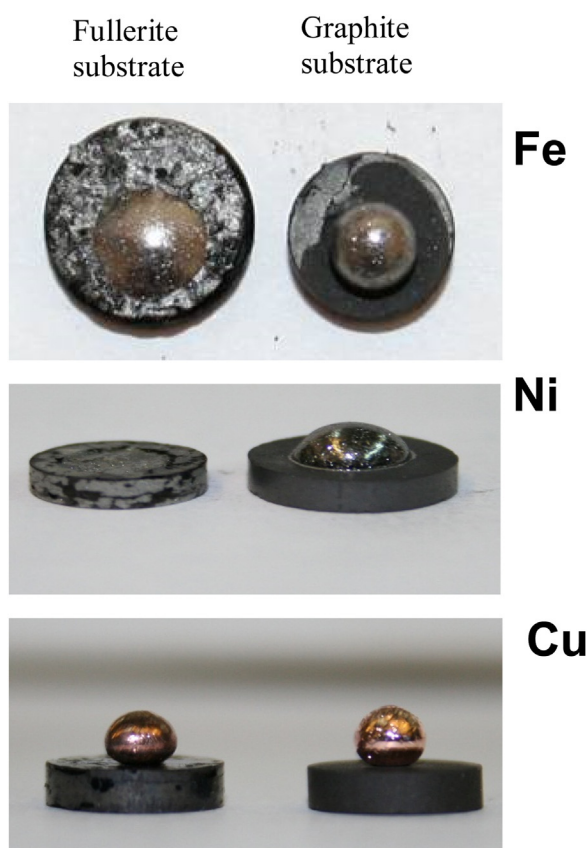


Fig. 3 – General view of samples after experiment. Heating temperature of Fe and Ni – 1500 °C, Cu – 1300 °C.

the size of the Cu drop remained unchanged. Let us consider the special features of the fullerite and graphite wetting by compared metals in more detail.

Contact interaction Fe-C. In Fig. 4 we can see considerable differences in the contact angles of the Fe melt on the graphite and fullerite substrates, which illustrates the dependence of the wetting angle on exposure time under certain temperatures for both carbon substrates. Different tendencies of the contact angle change are observed: the contact angle increases

on graphite and decreases on fullerite. Also, histograms are presented that illustrate the differences between the contact angle θ at the Fe-fullerite and Fe-graphite interfaces at 1300 °C. The same tendency is observed at other temperatures. Here one needs to understand that for reactive couples and / or porous substrates the parameter 'contact angle' is not enough to study the contact interactions.

Since iron has a high affinity for carbon, the interaction of the molten metal with the carbon substrate occurs at a high rate. The metal-carbon interface rapidly changes its physicochemical properties. With the increase of temperature and interaction time the horizontal line for determining the angle θ changes its profile. That is, the absolute values of the angle θ at the Fe-carbon interface become invalid; therefore, θ cannot be used as a criterion for assessing the wettability of a carbon surface by a melt. However, this characteristic is frequently used by different authors in the analysis of the wettability of carbon by an iron melt [8,9,38,39]. In our case, the observed different tendency of changing of the angle indicates a different nature of the contact interaction of fullerite and graphite with an iron melt. Presumably, we observe different kinetics of their interaction.

The differences between the equilibrium contact angles of melted iron on the graphite and fullerite substrates can be due to their different porosity. Calculations of porosity with account for volumetric extension coefficients of the materials β ($\beta_{\text{graphite}} = 8.76 \cdot 10^{-5} \text{ K}^{-1}$, $\beta_{\text{fullerite}} = 6.1 \cdot 10^{-5} \text{ K}^{-1}$ [36]) demonstrated that at 1300 °C the porosity E of graphite and fullerite made up 36.7%, and 17.4%, respectively. The experiments on annealing the graphite and fullerite substrates were undertaken at 1000 °C and 1300 °C (heating without metal). The initial porosity at room temperature made up $E_{\text{room}} = 29.6\%$ for graphite, and $E_{\text{room}} = 13\%$ for fullerite. After annealing at 1000 °C the porosity of the graphite substrate remained practically unchanged and made up $E_{1000 \text{ °C}}(\text{graphite}) = 29.9\%$, while in case of the fullerite substrate the porosity more than tripled: $E_{1000 \text{ °C}}(\text{fullerite}) = 42\%$. After heating to 1300 °C their porosity remained on the same level.

X-ray studies showed that such a considerable increase of porosity of the fullerite substrate was due to the structural changes in it. After high-temperature heating an X-ray amor-

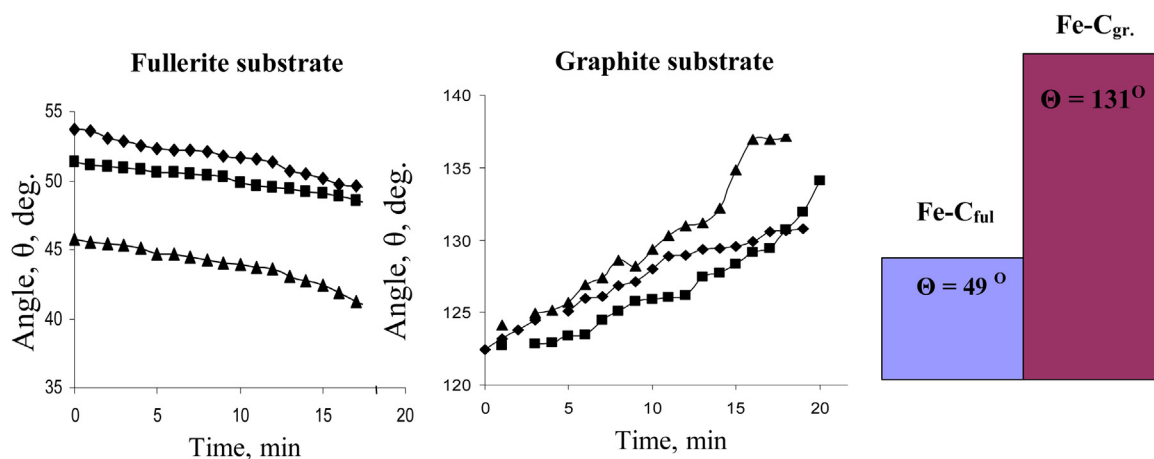


Fig. 4 – Contact angle as a function of time exposure for Fe melt on graphite and fullerite substrates at different temperatures: ♦ – 1300 °C; ■ – 1400; ▲ – 1500 °C. Histograms - contact angles, θ , at heating temperature 1300 °C

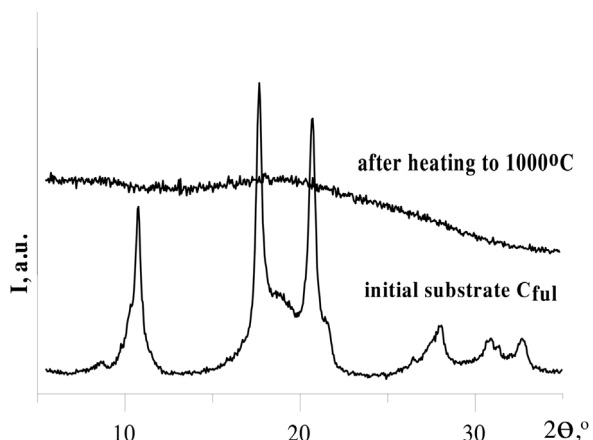


Fig. 5 – Structural changes in fullerite substrate at heating temperature 1000 °C

phous halo appeared on the diffractogram – an evidence of the fullerite destruction at as low as 1000 °C (Fig. 5). According to [40,41], ultimate temperature at which fullerite C_{60/70} remains stable is 850 °C. It thus follows that in the experiments on contact angle determination the metal melt interacts not with

fullerite, but actually with soot. This explains a better wettability of the initial fullerite substrates by Fe (soot has larger porosity than graphite: 42 and 29.9, respectively). The effect of the porosity of the substrate on the contact interaction Me-C was mentioned in [42]. In particular, in these papers, it was shown that the porosity of the carbon substrate determined the kinetics (the speed) of contact interaction in the Al-Si-C system. Graphite, vitreous carbon, pyrocarbon, and pyrographite were used as carbon substrates.

The structural changes in metal after the contact interaction of iron melt with a carbon substrate are evidenced by metallographic analysis data. Fig. 6 demonstrates metallographic analysis data for the iron samples heated to 1450 °C. The microstructure across the section of the whole drop was characterized by the presence of α-Fe, precipitated graphite (flaky, spheroidal, and vermicular graphite), flaky cementite and pearlite (α-Fe + Fe₃C). In this case the observed differences between the systems Me-graphite and Me-fullerite are manifested only in the quantity of graphite inclusions (for the Me-fullerite system their number is insignificantly higher). A lighter area is observed close to the graphite inclusions, which corresponds to α-Fe; no pearlite is present. That means that the metal structure in the areas indicated is depleted in carbon.

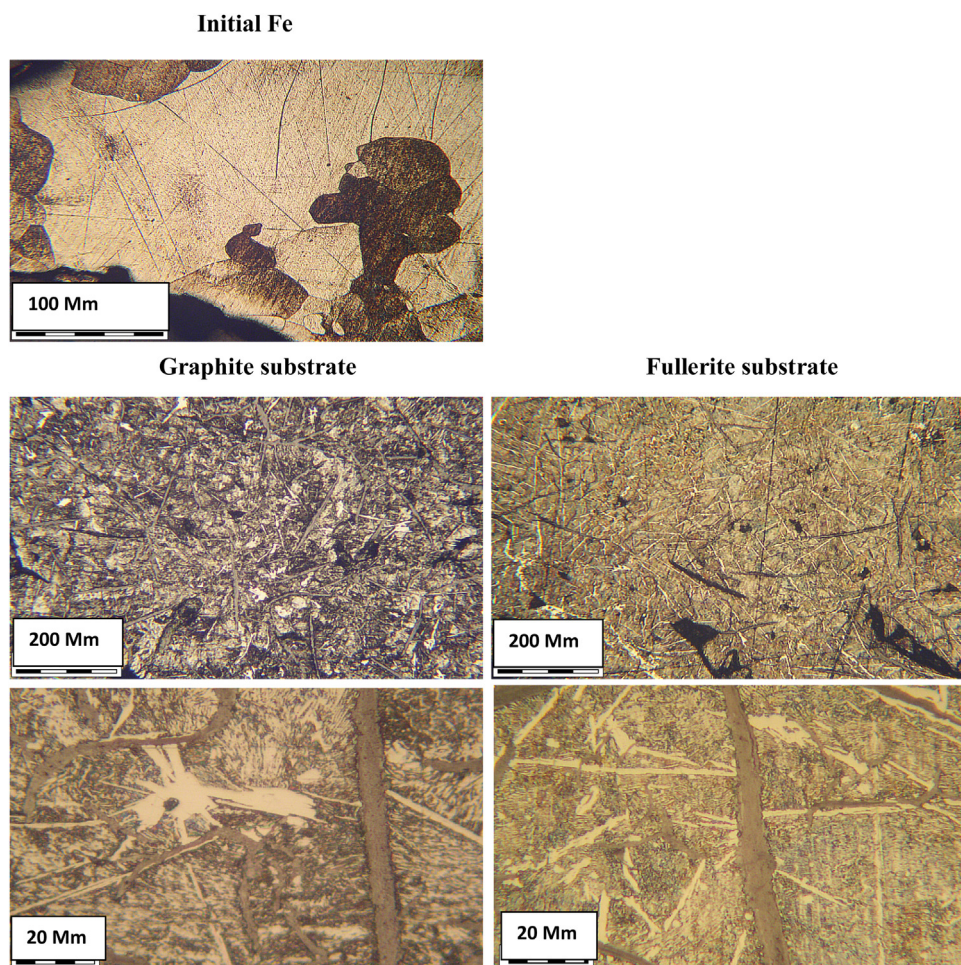
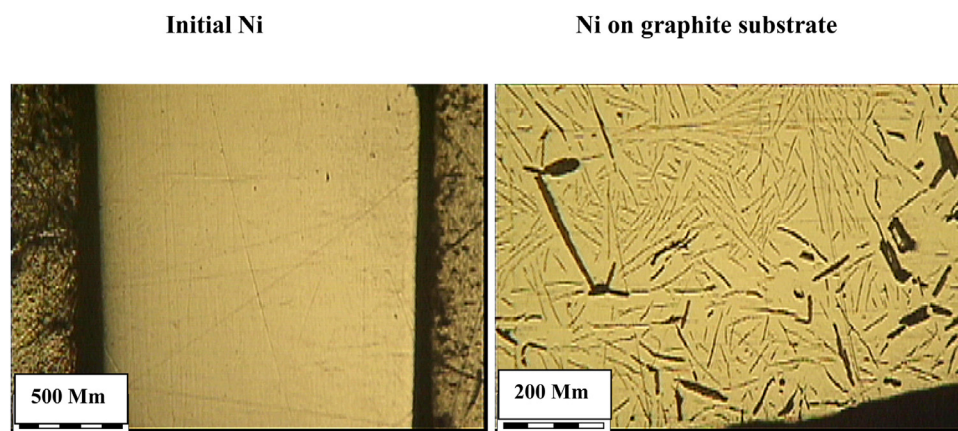


Fig. 6 – Structural changes in Fe at contact interaction with graphite and fullerite substrates (after etching). Temperature of heating – 1450 °C.

Table 1 – The change of the adhesion work, W_a for the melts Fe at different temperatures.

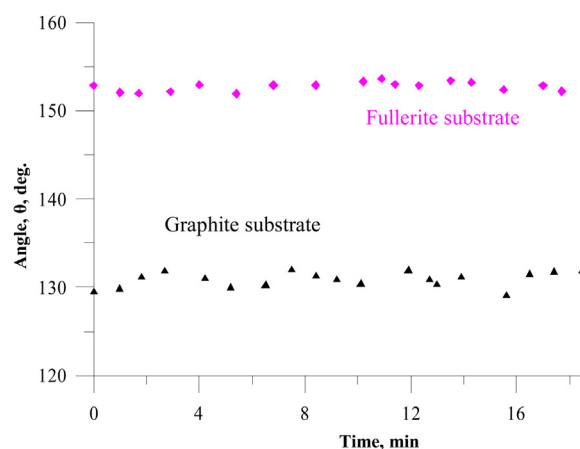
Temperature, $^{\circ}\text{C}$	Θ , deg	Fullerite substrate	W_a , J/m ²	Θ , deg	Graphite substrate	W_a , J/m ²
1250	-	-	-	-	-	2.14
1300	49	3.01		131		0.62
1400	48	3.04		134		0.56
1500	14	3.59		139		0.46

**Fig. 7 – Structural changes in Ni at contact interaction with graphite substrate.**

To account for adhesive interaction we performed the calculations of the work of adhesion W_a of a liquid to a solid. This work is required to form a liquid-solid interface: $W_a = \sigma_{ls}(1 + \cos\theta)$, where σ_{ls} is surface tension of liquid metals at the interface with gas and is a constant value. For iron it makes $\sigma_{ls}(\text{Fe}) = 1.82 \text{ J/m}^2$ [9]. As noted above, the absolute values of the angle θ at the metal-carbon interface are invalid. Nevertheless, calculations were performed for a qualitative assessment of the adhesive interaction at the Fe – C interface. In Table 1 presented are the calculated data on the change of work of adhesion W_a . It can be seen that in the case of the Fe-graphite interaction the work of adhesion essentially decreases (~5 times) with the rise of temperature. This evidences the considerable changes in the iron structure. Lower values of θ , and, accordingly, larger W_a , are indicative of a larger work of adhesion at the liquid-solid interface. The latter is presumably caused not only by the chemical interaction Fe-C, but also by a larger penetration (mechanical interaction) of the metal melt into the fullerite substrate (soot at interaction temperatures).

Contact interaction Ni-C. In contrast with Fe, full penetration of Ni melt into the fullerite substrate takes place (soaking), while into the graphite substrates Ni penetrates only partially. The metallographic analysis data show co-existence of just two phases – nickel and graphite (Fig. 7) as a result of the diffusion processes during the melt carbonization; no carbide phase was detected. Nickel does not form stable carbides at high temperatures, it dissolves carbon, and then, when Ni solidifies, it “expels” it as graphite [43].

Contact interaction Cu-C. There are some differences in the behavior of copper at high-temperature interaction with carbon substrates, as opposed to iron and nickel (See Fig. 1). Even with account for the different substrate porosities at melting temperatures of copper the metal does not penetrate

**Fig. 8 – Difference between contact angles θ at heating temperature 1300°C for Cu-C_{gr} and Cu-C_{ful} systems.**

into the substrate in both cases – with fullerite and graphite. After melting of Cu we observe the formation of the equilibrium contact angle - the contact angle does not suffer any changes in the process of isothermal exposure (Fig. 8). Contact angles of Cu on the graphite and the fullerite substrates much exceed the similar angles of Fe and Ni. It can thus be concluded that copper, being inactive to carbon, does not wet carbon substrates. The metallographic analysis of copper powders showed that the copper structure before and after the contact heating with carbon substrates remained unchanged; no graphite particles were observed. However, differences in the contact angle values were detected in the experiments with the graphite ($\theta \sim 152^{\circ}$) and the fullerite ($\theta \sim 130^{\circ}$) substrates. This can be explained by the different roughness of the initial substrates (see Fig. 2, the Ra value of the surface of

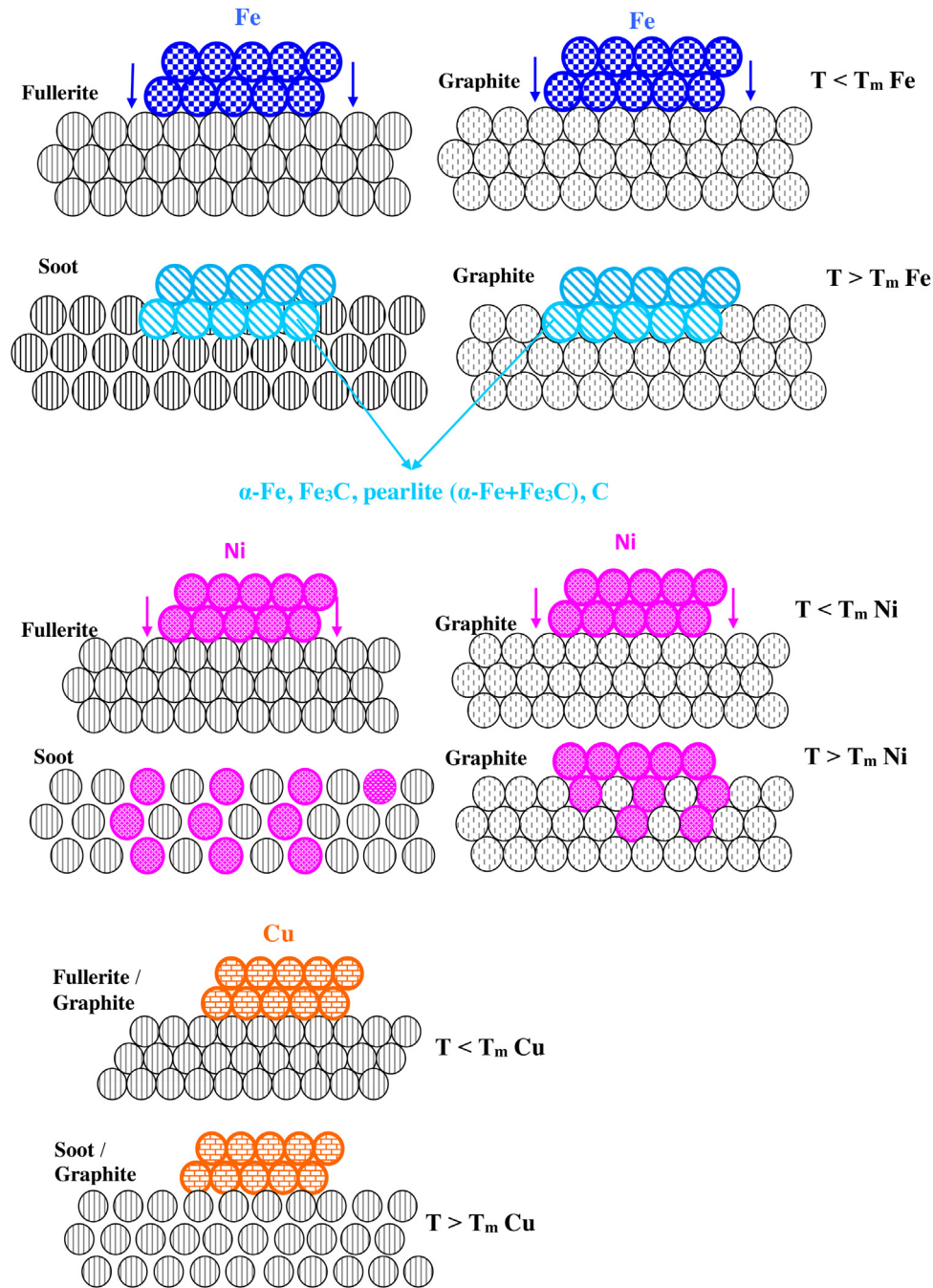


Fig. 9 – Scheme of contact interaction of Fe, Ni, and Cu melts with fullerite and graphite substrates with account for the change of their porosity.

Table 2 – The change of adhesion work W_a for Fe and Cu melts.

Me	W_a at T_m , J/m ²	W_a at T_{exp} , J/m ²
Fe	2.14 (1250 °C)	0.46 (1500 °C)
Cu	0.44 (1050 °C)	0.44 (1300 °C)

the fullerite substrate is ~ 3 times less than that of a graphite substrate). It is known that in the absence of wetting ($\Theta < 90^\circ$) an increase in roughness leads to an increase in the wetting angle [44–46].

Thus, the results of the investigation performed demonstrate different mechanisms of the contact interaction of the three melts - Fe, Ni, and Cu - with graphite and fullerite. Fig. 9 schematically presents the detected differences between the wetting of the graphite and the fullerite substrates by the metal melts. In the case of the Fe-graphite and Fe-fullerite systems considerable carbonization of the metal is observed in both cases with the formation of carbon-containing phases. As a result, the metal structure changes to form pearlite, insignificant quantities of cementite and graphite (Fig. 9a, in blue highlighted is the result of the structural changes). The dif-

ferences between the compared substrates concern only the quantitative ratio of the structures formed. Wetting of carbon substrates with liquid nickel probably occurs with the formation of unstable carbides with subsequent decomposition (b) under cooling conditions. Different degrees of wetting of the compared carbon substrates (soaking) can probably be explained by their different roughness and porosity. For copper, as opposed to Fe and Ni, no contact interaction with the carbon substrates occurs, even with account for the change of porosity in the initial fullerite substrate (Fig. 9c).

To confirm that the considered metal melts have different mechanisms of the contact interaction between the metals and the carbon substrates the work of adhesion W_a - which is a characteristic of the adherence between solid and liquid - was calculated. Table 2 contains the calculated data on the change of work of adhesion (for the system Me-graphite). It is seen that the work of adhesion essentially decreases with the increase of temperature of iron heating in the range of 1250 °C–1500 °C – from 2.14 to 0.46 (~ 5 times), which is indicative of considerable changes in the system Fe-C with temperature rise. The contact interaction in this system is a result of the chemical interaction that eventually leads to the structural changes in the metal. As for nickel. Liquid nickel dissolves carbon, which should lead to the typical Ni-C eutectic microstructure and its melting at a temperature significantly lower than the melting of pure nickel. Therefore, the Ni-C interface cannot be flat (a crater must be formed on graphite under a drop), and the contact angle measured by us is unreliable (therefore, the value of W_a is not given in the table). In case with copper this value remains at the level of 0.44 due to the metal inactivity to carbon.

In conclusion, we note that in this paper we have presented direct experimental data on the wetting of fullerite and graphite substrates by melts of pure metals Fe, Ni, and Cu. The information was obtained under the identical methodologic conditions. This indicates the reliability of the data obtained, which shows that fullerite and graphite, depending on the type of metal, are wetted by the melts in different ways. Obviously, further research is required to identify the nature of these differences.

4. Conclusion

For the first time ever the wettability of fullerites by Fe, Ni, and Cu melts has been studied, and the comparative analysis of the possible contact interaction at the metal-fullerite and metal-graphite interfaces has been carried out.

- 1 It has been shown that as a result of the contact interaction of Fe with fullerite and graphite, due to significant metal carburization, carbon-containing phases are formed. Metal structure changes to form a substantial amount of graphite, pearlite, and insignificant quantities of cementite. The differences in the structure of the final product between the compared carbon substrates, manifest themselves in slightly different quantitative ratios of the structural components.
- 2 In contrast with Fe, in the case of Ni the metal melt totally penetrates into the fullerite substrate and only partially into

graphite. Due to diffusion processes, Only two phases co-exist as a result of diffusion processes – nickel and graphite. No carbide phase is formed after cooling the melt.

- 3 No contact interaction of Cu melts with carbon irrespective of its modification (fullerenes, graphite) has been observed. The metal preserved its structure.
- 4 Different degree of wetting of the fullerite and graphite substrates by the metal melts is explained, mainly, by the amorphization of fullerite at heating temperatures higher than 1000 °C. Amorphization leads to an increase in the porosity of the carbon material.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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