

Measurement of actual temperature for a polylayer graphene film on a metal



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ABSTRACT

It is known that graphene layers, even thin, can lower strongly the temperature of heated metals. Pyrometry allows one to measure only brightness temperature, and the actual temperature remains unknown, although it is of interest. We propose a method to measure the actual temperature of surface graphene based on simultaneous measurement of thermionic emission and surface thermoionization of non-readily ionizable atoms, so called TESI method. It was used to measure first the change of actual temperature in layer by layer growth of graphene and graphite at a metal surface from the 1st layer up to ≈ 40 layers (≈ 12 nm), when the emissivity is due only to the film features.

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1. Introduction

It is known that carbon precipitation from metal bulk to the surface results in graphene and graphite growth which are studied intensively in recent years [1–4]. Growth of such a film on a metal surface induces strong decrease in brightness and actual temperature even if heating power is constant [5–8]. For example, graphite growth on Rh(111) with the thickness of 80–100 layers (≈ 34 nm) decreases the brightness temperature down by $\Delta T_B \approx 300$ K, and the actual temperature to ≈ 400 K (1). This results from increase of the emissivity ε : from $\varepsilon \approx 0.3 \div 0.4$ typical for Rh, Re or Mo, to $\varepsilon \approx 0.8 \div 0.9$ typical for bulky graphite [9,10].

Our recent paper [11] describes an interesting and unusual effect: surface graphene does not absorb light up to the thickness of ≈ 10 layers in contradiction with generally accepted concept that light absorption factor is about 0.5% for a single graphene layer [12–14]. To study the effect in detail, one needs a correct method to measure the actual temperature during the film growth.

Traditional methods do not fit totally the task features. Contact methods provide local cooling due to heat runaway, and can pollute the surface at a high temperature. Multi-wavelength pyrometry requires accurate data on emissivity at various wavelengths, and the result is very sensitive to variation in these constants. One also cannot use the temperature dependence of electric resistance: graphite growth is due to atomic carbon moving from the substrate bulk to the surface, and this process affects the sample resistance strongly.

Earlier, we have measured actual temperature only for initial and final growth stages, but not for the whole range [11]. The reason is that the film work function is changed with its thickness, and thermionic measurements do not work directly. This paper is aimed to develop such a method and to apply it to a polylayer graphene and graphite film on Re (10–10).

2. Experimental

Experiments were done in a high resolution, $\Delta E/E \approx 0.1\%$, UHV Auger spectrometer with the pressure of $1 \cdot 10^{-10}$ Torr; it was equipped with an additional unit for thermionic emission, surface thermoionization and probing the surface with CsCl molecular beam to measure the relative area of graphene islands on metal [8,15].

The samples were grain-oriented Re ribbons measuring $50 \times 1.5 \times 0.02$ mm with (10–10) face on the surface. They were uniform with work function, $e\phi = 5.15$ eV, orientation degree of (10–10) face was 99.9% according to X-ray diffraction. Sample purification and texturing were standard [16].

For carbonization, benzene vapor has been used at pressure $P_{C_6H_6} = 1 \cdot 10^{-5}$ Torr and carbonization temperature $T_C = 2050$ K. Growth of continuous graphene layer on the surface stops carbonization automatically, as molecular C_6H_6 does not dissociate on its surface, and atomic carbon ceases to penetrate into the metal bulk [11]. High resolution electron Auger spectroscopy (EAS) shows that carbon layer is actually graphene, and its thickness is one layer [5,15,17]. Probing the surface by molecular CsCl shows that the film is continuous; STM measurements support graphene structure and film uniformity [18].

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Brightness temperature has been measured by optical micro pyrometer at the wavelength 650 nm with the accuracy of ± 5 K. The ribbon was uniform with temperature at the length of 40 mm; thermoelectrons and ions were taken from its central part 5 mm long. Purity of Na and CsCl fluxes was tested using mass-spectrometry; main admixtures were K and Cs and their content was less than 0.1% for both fluxes.

To get graphene or graphite film thicker than one layer, the temperature has been decreased. This oversaturates the sample, and surplus carbon is precipitated onto the surface in a form of graphene [5, 15, 19]. The film thickness depends on the temperature $T < T_C$ and annealing time, layer-by-layer growth mode has been observed as in [16, 19]. Carbon precipitation kinetics at 1300–2000 K is controlled by bulky carbon diffusion for Re [15, 19]; if one knows the solubility limit, its temperature dependence, and activation energy, he can estimate the film thickness.

3. A method to measure the actual temperature

The method is based on simultaneous use of thermionic emission and surface thermoionization of non-readily ionizable atoms, called TESI in [20]. To solve our task, one needs to exclude work function from standard TESI formulas, as it depends strongly on the film thickness especially at the initial stages.

The thermionic current from a uniform emitter agrees with Richardson formula [21]:

$$I^- = A_R \cdot (1-R) \cdot S \cdot T^2 \cdot \exp[-\varphi/kT] \quad (1)$$

where A_R is the Richardson constant, for graphite it is $A_R = 150 \text{ A/m}^2 \cdot \text{K}^2$, S is the surface area, R is reflection factor for thermoelectrons, as a rule $R = 0$; k is the Boltzmann constant; T is the actual absolute temperature, and φ is the surface work function. I^- depends strongly on φ and drops with its growth.

If one probes the surface by atomic flux with ionization potential V , the ion current from the surface is determined by Langmuir–Saha equation if $V - \varphi \gg kT$ [20]:

$$I^+ = e \cdot \nu \cdot S \cdot A \cdot \exp[-(V-\varphi)/kT] \quad (2)$$

where e is the electron charge, ν is the incoming atomic flux, $A = Q_0/Q_+$ is the ratio of partition functions for neutral and ionic states of the probing atom, which is nearly constant for alkaline. I^+ grows with work function contrary to the thermionic current.

Both currents were measured in turn by a special collector system [16, 17]: a graphite film was grown to measure I^- versus growing time; then graphite has been dissolved and the sample has been heated at 2100 K for ≈ 1 min to provide uniform carbon distribution in the bulk. Then the experiment was repeated, but now I^+ was measured. The kinetics repeatability was very high, better than 1% at each time point, so the total precision was high also.

Fig. 1 presents changes in thermionic current and Na ion current ($V_{\text{Na}} = 5.14 \text{ eV}$) with graphite film thickness on Re (10–10); the initial state is a continuous graphene layer ($k = 1$) and the sample heating power is constant. One can see that initially, the thermionic current is decreased while ion current is increased; the brightness temperature is constant. This results from growth of the work function from $\varphi = 4.25 \text{ eV}$ for the first graphene layer on the metal up to $\varphi = 4.75 \text{ eV}$ at the thickness of $k = 8 \div 10$ layers, which is the same as for a basal face in a graphene single crystal [22]. At higher thicknesses $\varphi = 4.75 \text{ eV}$ is constant, but the thermionic current proceeds to be decreased, and the ion current is decreased also (region B in Fig. 1). This means that the actual temperature starts to be decreased, and the brightness temperature also starts to be decreased at this thickness range. This effect is due to the increase in heat emitted by the surface, i.e. the increase in sample emissivity, as the sample heating power is constant.

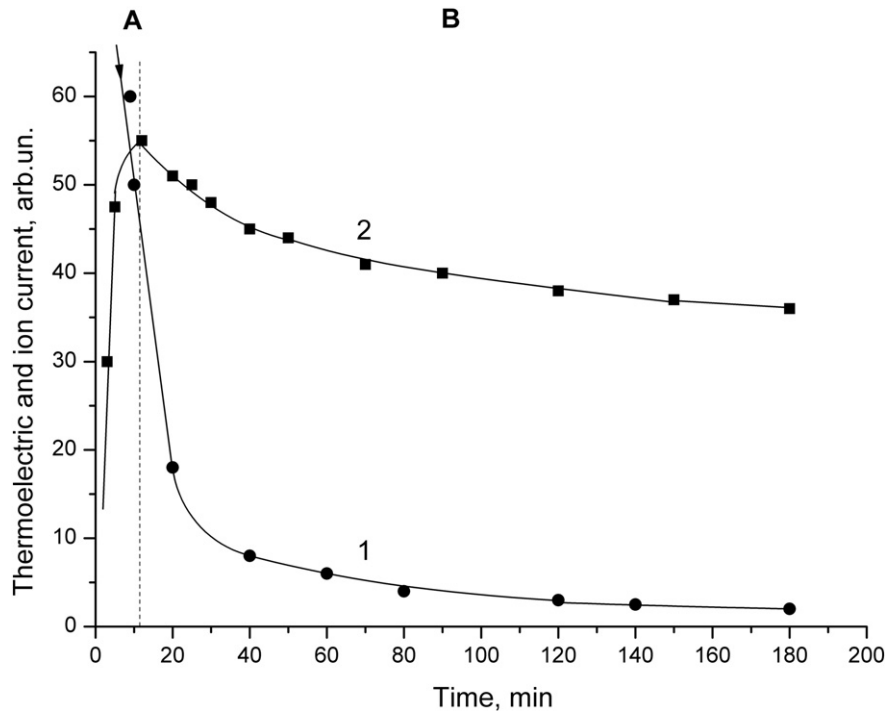


Fig. 1. Thermionic current (1) and Na^+ ion current (2) as functions of annealing time in graphene film growth on Re(10–10). The initial stage at $t = 0$ is a graphene single layer; initial temperature 1550 K, the sample heating power was constant. The sample carbonization temperature was 2050 K. A and B are regions of different temperature behavior. 60 arb. un. is equal to $3.2 \cdot 10^{-8} \text{ A}$.

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