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Short communication

Heating-induced variations of secondary electron emission from ion-cleaned copper samples

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ABSTRACT

Secondary electron (SE) emission due to electron impact depends strongly on surface conditions. The variations of SE yield and spectrum with the heating temperature of Ar-ion-cleaned oxygen-free copper samples are therefore measured in situ in a multifunctional ultrahigh vacuum system. The SE yield and the SE spectrum are observed to increase and to narrow, respectively, after sample heating. The maximum SE yield increases from 0.97 before heating to 1.25 after heating at ∼313 °C, and the corresponding full width at half maximum of SE spectrum decreases considerably from 9.3 to 5.5 eV. More CO_2 and Ar ions are shown to desorb at a higher heating temperature by residual gas analysis, indicating their contribution to the reduction in work function and surface potential barrier. Ar-ion desorption appears to affect the SE spectrum more than the SE yield. The obtained results provide a new insight into complicated surface influences on SE emission in thermal applications of scanning electron microscopy.

1. Introduction

Strongly depending on surface conditions, SE emission (SEE) from sample surfaces due to electron impact has always been a key factor in scanning electron microscopy (SEM) (Reimer, 1998; Seiler, 1983). Here, a sample surface is easily contaminated by adsorbed gases and beam-induced hydrocarbons (Egerton et al., 2004; Mullerova and Frank, 2003). Serious surface contamination alters the work function and degrades the SE imaging contrast and resolution (Cazaux, 2012; Jepson et al., 2010, 2011; Rodenburg et al., 2010; Sealy et al., 2000; Soong et al., 2012). It is known that water vapor and carbon or CO_2 can intensify and suppress SEE due to lower and higher work functions, respectively (Cazaux, 2011; Cimino et al., 2012; Hilleret et al., 2003; Kuzucan et al., 2012; Zhang et al., 2014).

Although widely used to remove surface contaminations, Ar-ion cleaning may form derivatives such as carbon or CO_2 (Holm and Storp, 1977) and naturally leave positive ions at a cleaned metal surface, also affecting SEE properties (Catalfano et al., 2013; Everhart et al., 1976; Jenkins and Chung, 1971; Joy et al., 2004). Furthermore, for some SEM sample treatments and observations and other applications in thermal environments (Dowell et al., 2006; Homma et al., 1996; Okayama et al., 2005; Wu and Hull, 2013), surface properties and resultant SEE characteristics of metal samples should be changed because of thermal desorption (Bojko et al., 2000; Zhang et al., 2014). It is therefore important to know in advance the SEE variations due to different surface states and observation conditions. However, detailed information is extremely lacking about the heating-induced SEE variations of ion-cleaned metal samples.

We have thus explored the dependences of the SE yield (SEY) and the SE spectrum (SES) on the heating temperature for ion-cleaned oxygen-free copper (OFC) samples. In a multifunctional ultrahigh vacuum (UHV) system, the OFC samples were cleaned by Ar ions and then heated at different temperatures. Residual gas analysis (RGA) was used to monitor surface desorption during the heating process. SEY and SES were measured in situ both before and after heating. In this paper, after introducing sample treatment and SEE measurement methods, we present the measurements of SEY and SES, variations of the maximum SEY and full width at half maximum (FWHM) of the SES, and RGA results. In addition, the possible mechanisms of the heating-induced SEE variations are briefly discussed from the point of view of work function and potential barrier.

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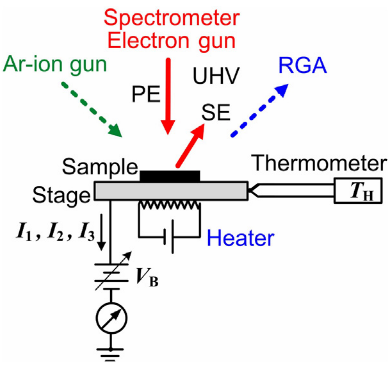

Fig. 1. Schematic of experimental set-up for sample treatment and SEE measurement in UHV. An OFC sample was heated after Ar-ion cleaning, while RGA was performed by a quadrupole mass spectrometer. The SEY was measured through electron currents I_1–I_3 of the sample corresponding to different bias voltages V_B of 500, −20 and 50 V, respectively. The SES was detected by a spectrometer (cylindrical mirror analyzer) integrated with the electron gun supplying the primary electron (PE) beam normally incident on the sample.

2. Experimental

Fig. 1 outlines the methods of sample treatment and SEE measurement, and some details of the related UHV experimental system have been described elsewhere (Zhang et al., 2012, 2014). The samples under study were OFC foils (CAS RN: 7440-50-8) with purity 99.95% and thickness 0.25 mm. They were cut into rectangles of about 1 cm × 1.2 cm and cleaned with anhydrous alcohol and de-ionized water. A prepared sample was inserted into the UHV chamber and cleaned by a suitable condition of Ar-ion with 1 keV for 10 min. The ion current density and incident angle were fixed at ~1 μA/cm^2 (current in ~3 μA and spot size in ~3 cm^2) and 45°, respectively. The ion-cleaned sample was then heated by a resistive heater with a changeable current for 2 h. The detected stage temperature reached its stable value T_H rapidly in ~10 min. The stable heating temperature T_S of the sample could be determined by the calibration relation $T_S = 0.77\,T_H + 13\,°\text{C}$ with the standard deviation of $\pm(0.016\,T_H + 4\,°\text{C})$ in the T_H range of 100–400 °C. RGA was carried out by a quadrupole mass spectroscope (Pfeiffer QME 220) during sample heating, in which gas desorption came mainly from the sample instead of the experimental system (Zhang et al., 2014).

SEE characteristics were eventually measured in the following way. First, SEY δ, the ratio between emitted true SEs and PEs, was obtained from the difference between total yield σ (Zhang et al., 2012) and backscattered electron (BSE) yield η (El Gomati et al., 2008) with respect to the PE energy E_{PE}. As shown in Fig. 1, different sample currents I_1, I_2 and I_3 were respectively obtained at different V_B of 500, −20 and 50 V, corresponding to the PE current, the sample current excluding true SEs, and the sample current excluding BSEs. Then, σ and η were determined respectively as $\sigma = 1 - I_2/I_1$ and $\eta = 1 - I_3/I_1$. This resulted in $\delta = \sigma - \eta - I_3 - I_2/I_1$.

The SES was detected at a fixed $E_{PE} = 300$ eV by the spectrometer (DESA 150 analyzer) vertically above the sample. The spectrometer takes advantage of a cylindrical mirror electrostatic fields and a large acceptance solid angle of 6% of 2π sr, with an energy resolution of less than 0.1 eV. The SES collection was lasted for ~0.5 min and no effect of contamination was observed since samples have been cleaned by both Ar ion and thermal treatment. Here, a negative bias, $V_B = -15$ V, was applied to the sample to collect SEs more effectively.

To avoid the possible PE influence on SEE, the PE currents were controlled constantly as ~10 nA and 0.6 nA for measurements of SEY and SES, respectively. The total irradiation doses were respectively kept below 10^{-5} C mm^{-2} and 10^{-7} C mm^{-2}. Note that we measured the SEY and SES at three different points on a same sample and the deviations of the maximum SEY, the FWHM and the most probable energy (MPE) of SES were 1.6%, 2% and 5.3%, respectively. Thus, we present the measured data of SEE from one of the test points in the following results.

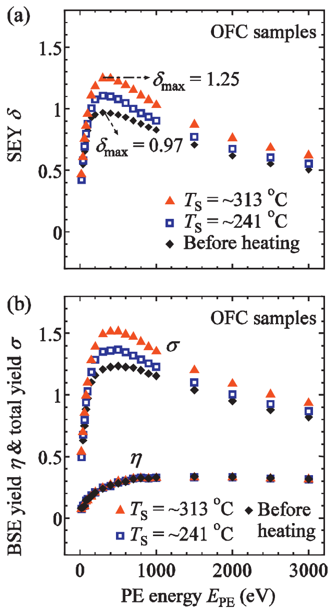

Fig. 2. Measured curves of (a) SEY and (b) BSE yield and total yield as a function of E_{PE} for two ion-cleaned OFC samples before heating and after heating at two different T_S, respectively. The SEY and the total yield are increased due to sample heating.

It should be noted that the heating current has to be cut off to isolate its electric influence on the SEE when the measurements were taken. The sample temperature should decrease a little bit during the measurement. Fortunately, this had little influence on the results since the effect of heat treatment will not be reversed with temperature decreasing (Zhang et al., 2014).

3. Results and discussion

3.1. Heating-induced SEE variations

Fig. 2(a) shows the measured SEY curves of ion-cleaned OFC samples before heating and after heating at different temperatures T_S. Apparently, increasing T_S can increase δ, especially around PE energy $E_{PE} = 300$ eV where the maximum SEY, δ_{max}, is shown. For example, δ_{max} increases from 0.97 before heating to 1.1 and 1.25 after heating at T_S = ~241 and ~313 °C, respectively. Here, the increase in δ_{max} indicates the reduction in work function (Walker et al., 2008). However, the increase in δ becomes gentle with increasing E_{PE}, and δ tends to that in the case before heating. Note that the heating-induced increase in δ is opposed to the case where a sample was not cleaned by Ar ions (Bojko et al., 2000; Hilleret et al., 2003; Zhang et al., 2014). In Bojko's work (Bojko et al., 2000), the maximum of total SEY decreases from 2.5 before bakeouts to 1.4 after 24 h bakeouts at 350 °C for technical copper. This should be correlated to the gas species desorbed during sample heating, which may increase or decrease the work function and therefore suppress or promote SEE. In addition, thermal desorption of Ar ions may reduce the positive potential barrier and therefore increase δ to some extent. For comparison, Fig. 2(b) illustrates different influences of T_S on BSE yield η (lower plots) and total yield σ (upper plots). Here, T_S hardly affects η, and σ behaves in a similar way with δ as shown in Fig. 2(a) as expected (Ding et al., 2004).

Fig. 3 presents the measured energy distributions of SEs from the ion-cleaned OFC samples before heating and after heating at different T_S. We can see that the SES varies more obviously due