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High Resolution Field Emission Spectra from Niobium (100) Tip Surface*

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We have developed a new apparatus to measure the energy spectra of field emission electrons with the high resolution. By using the apparatus, we have measured the high-resolution spectra of the electrons emitted from the Nb(100) tip at liquid-nitrogen temperature and detected the electronic surface (sub-surface) resonance states and the atomic vibrations of chemisorbed hydrogen.

KEYWORDS: Field emission, Niobium, Hydrogen adsorption, Coherent electron beam, Atomic vibrations

1. Introduction

Since Crewe *et al.* were the first to experimentally demonstrate the dramatic improvement in the resolution of transmission scanning electron microscopy by using a FE tip as an electron source in 1968,[1] the performance of the instruments such as scanning electron microscopes and analytical electron microscopes has remarkably advanced by improvement of electron optics system. However fundamental properties of electron sources have not been improved so much, and further progress in electron sources has been required for a long time.

Under such a circumstances, in 1969, Gadzuk theoretically showed the possibility of coherent electron beam utilizing field emission electrons from the superconducting tip, of which energy spread is estimated less than 0.05 meV.[2] Since then, several researchers have tried to confirm the coherent electron beam. However no one succeeded in detecting it because the energy resolutions of their electron spectrometer were too poor to observe the coherent electron beam.

Therefore, we have developed a new high resolution electron spectrometer to confirm it experimentally.[3] Moreover, in order to realize the field emission from superconducting tips, we have also improved the tip fabrication techniques of niobium (Nb), of which superconducting critical temperature, 9.2 K is the highest among the simple metals.

On the other hand, with regard to not only applications to electron sources but also the fundamental research, field emission phenomena have been extensively studied by many researchers. The total energy distribution of field emission electrons yields two types of spectroscopic data of the surface. The structure in the total energy distribution induced by the resonance tunneling reveals the local density of states at the surface. The structure induced by inelastic tunneling yields the characteristic energy losses of the atomic vibrations of the adsorbates and surface atoms. By means of measuring field emission spectra, we can obtain two kinds of information on the surface at the same time.

In this paper, we report on the field emission electron spectra from Nb(100) tip measured by the newly developed high resolution electron spectrometer. Since we carried out experiments at room temperature and liquid-nitrogen temperature, superconducting states of the Nb tips have not yet been realized. However, we obtained several interesting information on Nb(100) surface, which is the evidence that the energy resolution reaches higher region than conventional ones.

2. Experimental apparatus

For a few years, we have tried to improve the performance of our apparatus. The apparatus is mainly consist of three parts; vacuum system, FE gun and electron spectrometer. The characteristics of each part are described below.[3]

(a) *Vacuum system*

In order to obtain XHV condition easily, our main chamber is made of special stainless steel (Clean Z, NKK), and evacuated by the oil diffusion pump with a liquid nitrogen cold trap and the titanium sublimation pump with liquid nitrogen cold shroud. After 20h baking at 200°C, UHV condition of 5×10^{-9} Pa is easily realized. Moreover, with liquid helium in the cryostat, the pressure reaches below 1×10^{-10} Pa which is the measurable limitation of the extractor gauge.

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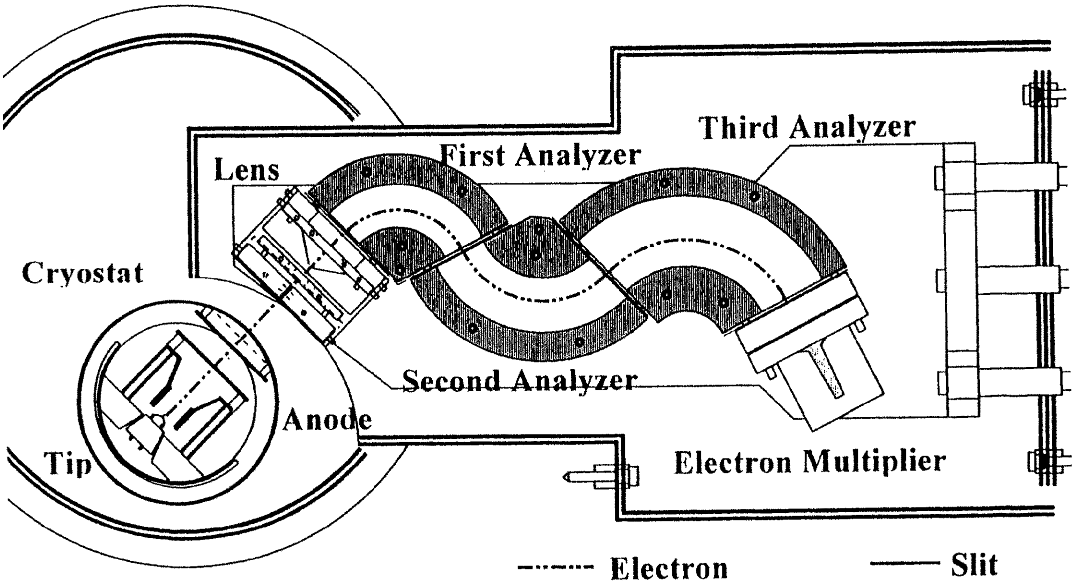

Figure 1. The schematic diagram of high resolution electron spectrometer

(b) ***FE gun***

FE gun is consist of a florescent extract electrode, four deflecting electrodes and specimen of a sharp tip. It is mounted on the liquid helium tank of cryostat, and surrounded with thermal shield. Although the temperature low enough to realize superconducting states of Nb tips has not been obtained yet.

(c) ***Electron spectrometer***

Figure 1 shows the schematic diagram of high resolution electron spectrometer. In order to obtain high energy resolution, three stages of cylindrical electrostatic analyzers are adopted. The potentials of all electrodes of the spectrometer are provided by newly developed low-noise power supply.[4] They are computer-controlled. Moreover the spectrometer is triply shield by "permalloy". The residual magnetic field at the points of electron trajectory were below 1 mG. Using this spectrometer, we have already realized the energy resolution of 0.8 meV under the best condition.

3. Experimental procedure

The Nb tips were fabricated by electro-chemical etching in a solution of HNO_3, HF, H_3PO_4 and CH_3COOH.[5] After the field emission patterns of these tips were evaluated in the separated XHV chamber, only the tip exhibiting a beautiful symmetry pattern was mounted in the electron gun connecting with high energy resolution electron spectrometer.

Experiments were carried out in the XHV chamber at either room temperature or low temperature. During experiments, the pressure was kept below 5×10^{-10} Pa and the energy resolution of electron spectrometer was about 5 meV. The tip surface was cleaned by flash heating. In order to confirm the clean surface, we observed the field emission pattern, which was projected onto the florescent extract electrode in front of the tip. The electrode has a probe hole at the center. Only the electrons which pass through the probe hole can reach the electron spectrometer.

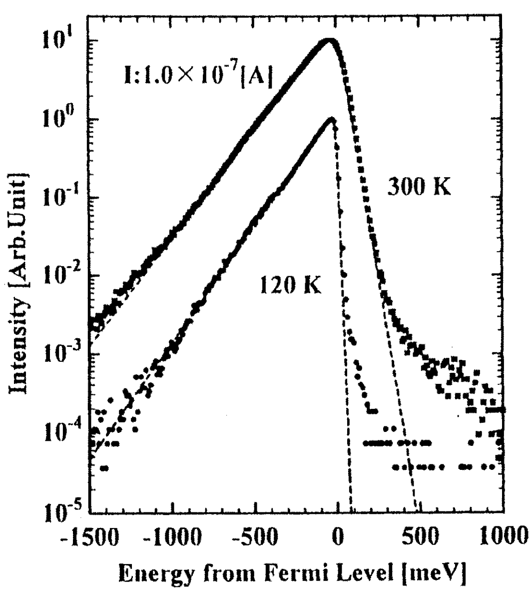

Figure 2. The temperature dependence of spectra (300K, 120K)

Therefore, we can select the observed surface area by tuning the voltages applied to deflecting electrodes. In this work, we chose the (100) surface to investigate because of the high brightness.

4. Results and discussion

Figure 2 shows the typical field emission electron spectra from the clean Nb(100) area at room temperature and low temperature, respectively. These spectra were measured just after cleaning. The emission current was 1.0×10^{-7} A. Dashed curves are the theoretical ones calculated on the basis on the Fowler-Nordheim (FN) theory.[6] Main parts of experimental curves agreed with theoretical ones. Therefore we can determine the tip temperature by fitting the parameter to reproduce the observed curves. Two temperatures in Fig.2 were estimated at 300 K and 120 K. In Fig.2, we found two large discrepancies between the experimental and theoretical curves on both high and low energy sides. These discrepancies were observed at both room temperature and low temperature.

Figure 3 shows the time dependence of spectra at low temperature. These spectra were measured just after cleaning and 18 hours later from cleaning. A hump appeared around -500 meV below Fermi level when the tip surface was clean. It had a tendency to disappear by long lapse even in XHV, which indicates that the hump originates from surface or sub-surface electronic states.

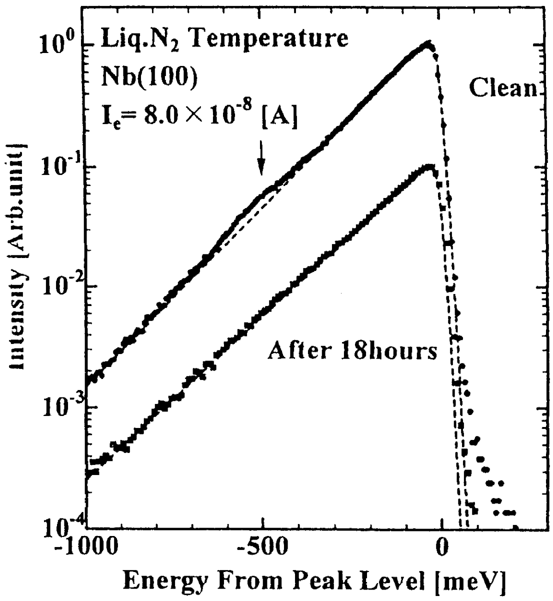

Figure 3. The time dependence of spectra (just after cleaning, after 18 hours)

Figure 4 shows FE spectra for various emission currents. There is a large discrepancy on high energy side between experimental data and theoretical ones, and it increases with increase of FE current.

Therefore, Figure 5 shows the relationship between total emission current I and the current in high energy tail i*. The extra current in the tail was proportional to the square of total emission current. This result supported the former theory by Lea and Gomer.[7]

Total energy distributions of field emission electrons from Nb (100) surface were measured as a function of the hydrogen exposure. Figure 6 shows a typical enhancement factor R in the hydrogen adsorbed process. They were the ratios of the measuring distributions to theoretical ones on the basis of FN theory. There are some interesting features remarked in Figure 6.

At first, when the tip surface was clean, a small shoulder appeared at -100 meV below Fermi level. Since it disappeared with increasing the hydrogen exposure, we assigned it a surface resonance state on Nb(100) surface.[8]

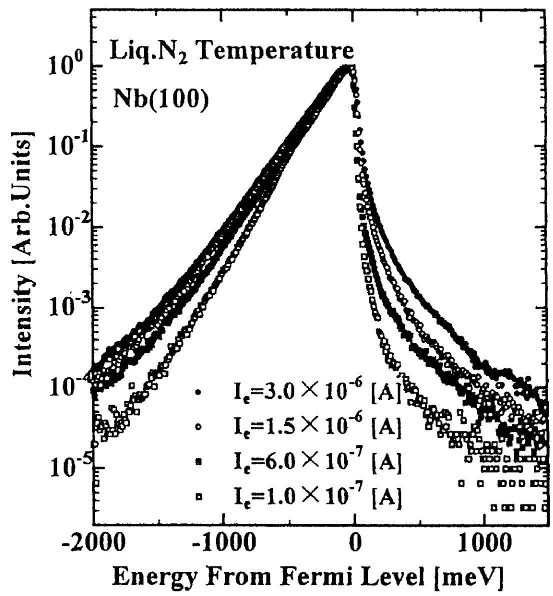

Figure 4. Field emission spectra for various emission currents

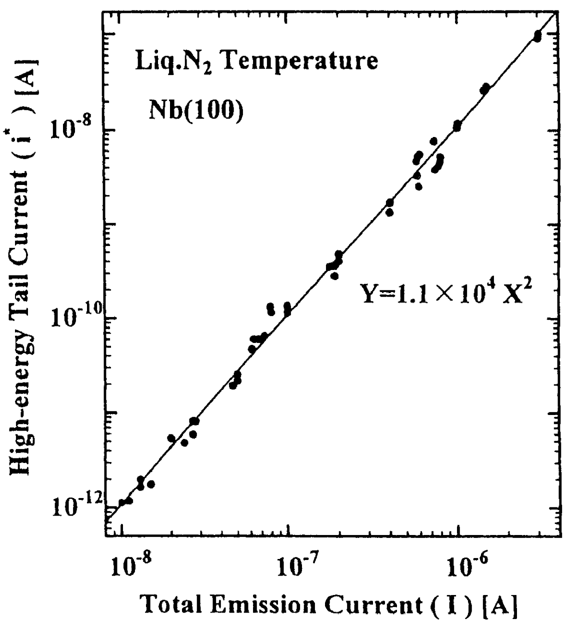

Figure 5. Total emission current (I) vs. High-energy tail current (i*)

Secondly, a new hump appeared around -150 meV with increasing the hydrogen exposure. We assigned it atomic vibrations of hydrogen adsorbed on the Nb (100) surface in the following reasons: It was observable only after the hydrogen was dosed, and the energy position of -150 meV below Fermi level corresponds to the frequency of 120 meV (the maximum position of energy distribution was -30 meV from Fermi level.), it agrees with the atomic vibration frequency of chemisorbed hydrogen on the Nb(100) surface measured by high resolution electron energy loss spectroscopy.[9]

Thirdly, a large hump around -500 meV was always observed even after the hydrogen exposure above 100 L, but this hump had a tendency to disappear after long lapse time in XHV. Therefore we tentatively assigned this hump to a

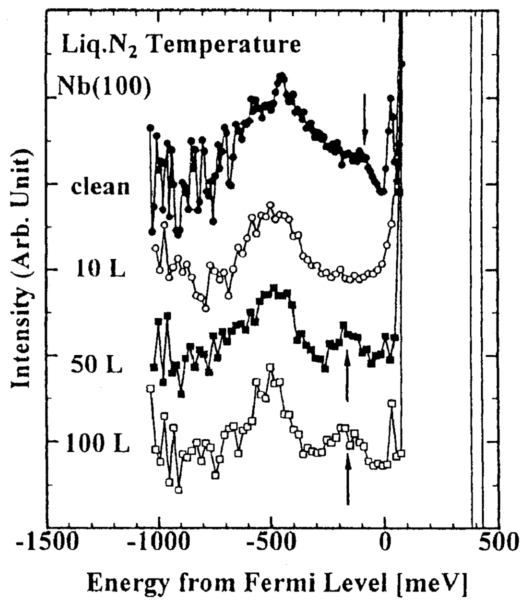

Figure 6. The enhancement factor R in the hydrogen adsorbed process

subsurface resonance state.[10] The disappearance presumably originates from the oxygen atom penetration to bulk and then the oxygen atoms come from carbon monoxides in residual gas.[11]

5. Summary

We investigated field emission from Nb(100) tip surface by using high energy resolution electron spectrometer, and obtained total energy distributions of field emission electrons. Despite of the excellent fit over most of range, experimental data had two large discrepancies from theoretical ones on both high and low energy sides. They result from electron-electron scattering in metal. Moreover we observed surface and sub-surface resonance states of Nb(100) surface and atomic vibrations of hydrogen adsorbed on Nb(100) surface.

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