

Gas field ion source from an Ir/W<111> single-atom tip

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We show that a thermally stable Ir/W<111> single-atom tip is a very good point ion source for rare gases (He, Ar) and reactive gases (H₂, O₂). The ion beams are emitted from the topmost atom with a very small opening angle (<1°) and, most importantly, they exhibit high brightness. In addition, the ion currents are very stable. These good properties together with the long lifetime of the tip and the reliable tip preparation method make this tip especially suitable for applications in gas field ion source focused ion beam systems. © 2008 American Institute of Physics.

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Currently, most focused ion beam (FIB) systems employ liquid-metal ion sources (LMISs), in which a liquid metal is supplied and field evaporated from a cusp above the liquid Taylor cone. Because of the simplicity and reliability of the source, the LMIS-FIB systems have gradually become an important diagnosis and modification tool for the semiconductor and nanotechnology.¹ Typical LMIS-FIB systems can achieve a resolution of 20 nm with a brightness of $\sim 10^9$ Am⁻² sr⁻¹. The resolution and the brightness are mainly limited by the relatively large virtual source size (50 nm), the large opening angle, and the wide energy spread of the ion beams (ΔE : 5–50 eV).² Another undesirable property is that liquid-metal ions are inevitably implanted into the sample, which may change the physical and chemical properties of the materials.

Gas field ion sources (GFISs) were once considered for FIB systems. They rely on the field ionization of the attracted molecules to the tip apex.³ Their virtual source size (~ 1 nm) and the energy spread (<1 eV) are at least one order of magnitude smaller than those of LMISs.⁴ In addition, the ion implantation problem can be avoided if a noble gas ion beam is used. However, GFISs have not been used in commercial FIB systems mainly because of their low angular intensity and poor reliability.

Gomer suggested that formation of a tiny protrusion on rounded field emitter might produce a lens effect and increase the beam intensity.⁵ This idea was later experimentally confirmed by Hanson and Siegel in gas field ion emission.⁶ Since 1988, Jovsten *et al.* had widely characterized the ionization behaviors and demonstrated significant emission enhancement from their so-called supertips.⁷ For the past two decades, other groups also have proposed various approaches to build up similar bump structures on the tip apex (a nanotip or an ultrasharp tip).⁸ However, the procedures are tedious, unreliable, and often require special facilities. The lifetimes of these tips may not be long because their structures are neither thermodynamically nor chemically stable. These problems along with the random orientation of the nanoprotusions still hinder their application in commercial FIB systems.⁹

In 2001, Fu *et al.* first demonstrated a Pd-covered W (111) single-atom tip (SAT) through vacuum deposition of an ultrathin Pd film on a clean W tip surface followed by thermal annealing.¹⁰ Later, Kuo *et al.* further simplified the preparation process by replacing the tip cleaning and the vacuum deposition with electrochemical processes and successfully prepared several different types of noble metal-covered W(111) SATs.¹¹ In contrast to other techniques, this type of SATs is thermally stable and chemically inert and, thus, can be regenerated through a gentle annealing if the apex is damaged. Most importantly, the stacking of the pyramidal nanoprotusion remains the same for each regeneration, which demonstrates its high reproducibility and reliability. Recently, these SATs have been shown to be highly efficient and reliable electron sources.¹²

In this letter, we report our measurements on the gas source ion emission characteristics of Ir/W SATs. Iridium, instead of other noble metals, is chosen because it can sustain higher temperatures, higher positive electric fields, and chemical attacks.¹³ Hydrogen, helium, argon, and oxygen ion beams are generated and characterized. The first two lightest ions yield the lowest sputtering rates which is beneficial for scanning ion microscopy.¹⁴ The argon ion has a large mass and can provide a high sputtering rate, suitable for ion milling. Due to the high secondary ion yields, an oxygen ion beam may have important applications in secondary ion mass spectrometry.¹⁵

The operation principle of GFIS is the same as in field ion microscopy,¹³ as illustrated in Fig. 1. The procedure for preparing an electroplating Ir/W <111> SAT has been described earlier.¹¹ Gases are admitted through leak valves. Since the ion current is in the range of 10^{-14} – 10^{-11} A, small currents are measured through amplification of a microchannel plate (MCP). The amplification gain is calibrated in the high current regime, where direct measurement without amplification is possible.¹⁶ Figure 1 shows a schematic diagram of a SAT apex and the corresponding He⁺ beam profile from an Ir/W SAT. Only one intense spot with a half-angle of 0.75° is seen on the MCP screen, which extends a field of view of $\sim \pm 50^\circ$ from the tip axis. This single spot indicates that emission occurs only from the topmost atom. This small source size and the small opening angle (compared with 25° for Ga-LMIS) are particularly favorable for achieving high angular intensity, high brightness, and low spherical aberration.

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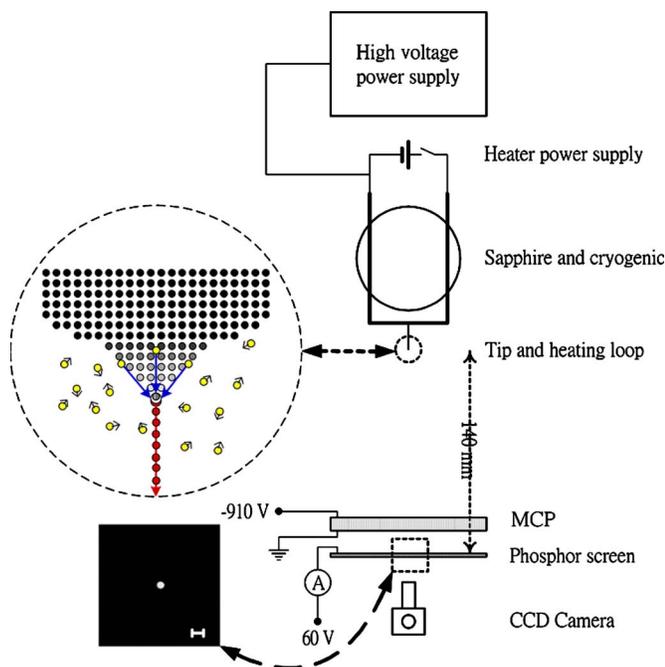


FIG. 1. (Color online) Schematic diagram showing the ion beam characterization system. The SAT with a supporting loop is mounted on a sapphire rod for good thermal conductivity and electric isolation. A schematic diagram of a SAT is shown on the left. Gas molecules (yellow) adsorbed on tip shank are attracted toward the apex (blue arrows) by the electric field and are ionized at the apex (red). The corresponding He^+ beam image is shown below with the scale bar equivalent to 2° from the apex.

tion, which are important characteristics for an FIB system.

Figure 2 shows the current stability of three different gas ion beams emitted from a Ir/W(111) SAT. Clearly, they are very stable with instability of 3% for the He^+ ion beam, 5% for the H_2^+ ion beam, and 7.7% for the O_2^+ ion beam. We have made similar measurements of the ion currents more than 20 times from different Ir/W(111) SATs which are either freshly prepared or regenerated through gentle annealing. Such steady beam currents without low frequency flicker noise are regularly observed. We note that the slightly higher instability for the O_2^+ ion beam is mainly due to the O_2 condensation on cryostat head, which makes our gas pressure control more difficult. Note that H_2 and O_2 are very reactive on most metals. Such reactive gases can enhance the removal of the tip surface atoms at high positive fields and, thus, shorten the lifetime of a nanotip.¹³ Most amazingly, the single-atom emission site of Ir/W(111) SAT is very stable and does not show any degradation under fields above 5 V/\AA after a total operation time of 80 h. We also find that the SAT can be regenerated for more than 50 times, therefore, its lifetime is long enough for commercial applications.

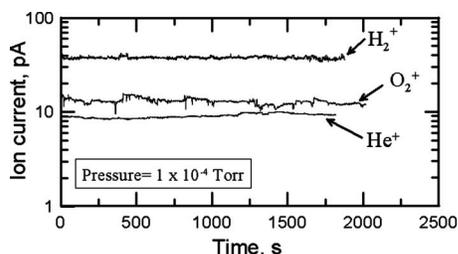


FIG. 2. H_2^+ , O_2^+ and He^+ ion beam currents as a function of time.

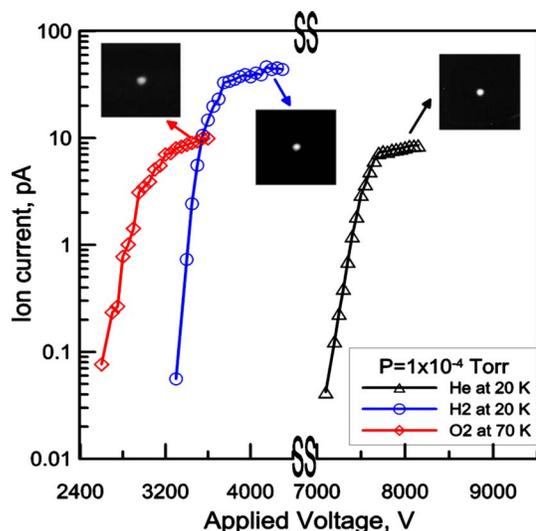


FIG. 3. (Color online) Currents vs voltage characteristics for ion beams field emitted from an Ir/W(111) single-atom tip.

To search for the optimum operation conditions for GFIS, several characteristics of the ions beams are measured. Figure 3 shows the I - V characteristics of three different gas ion beams. For each I - V curve, two different slopes can be seen, similar to the behavior of a normal hemispherical tip.¹³ In the low-field regime, the ion current increases steeply with the electric field. When the voltage is raised beyond a certain value, the current increases much more slowly and eventually reaches a plateau. The measurements show that the hydrogen ion beam is the brightest with a saturation current of 50 pA at 20 K.

It has been known that, in the low-field regime, the ion current is limited by the ionization rate of gas molecules on the tip apex.¹³ Thus, the current shows a strong dependence on the electric field. Above a certain high electric field, the gas supply from the tip shank to the tip apex becomes the rate-limiting factor; thus, a saturation ion current is reached. To get an ion beam with the highest brightness, a GFIS should be operated in the high field regime.

Figure 4 shows the temperature dependence of the saturation ion currents. Ar^+ ion beam current exhibits a maximum value at 72 K and decreases when the temperature deviates from this temperature. However, the He^+ current shows a strong increase with decreasing temperature down to 20 K, which is the lowest temperature reachable with our cryostat. The optimum temperature for the He^+ ion beam is probably just below 20 K.

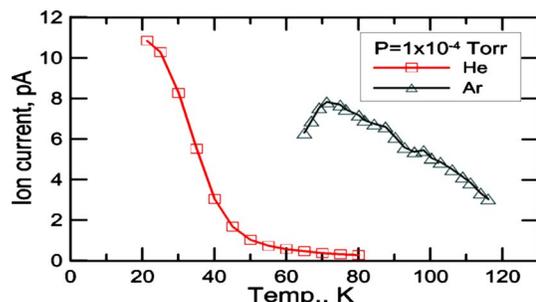


FIG. 4. (Color online) He^+ and Ar^+ ion beam currents as a function of the tip temperature.

TABLE I. Characteristic of various ion beams emitted from Ir/W <111> single-atom tip.

	He ⁺	Ar ⁺	O ₂ ⁺	H ₂ ⁺
Temp. at cryostat (K)	20	72	70	20
Current (pA at 1×10^{-4} Torr)	11	8	10	47
Angular intensity ^a ($\mu\text{A sr}^{-1} \text{Torr}^{-1}$)	170	130	160	760
Brightness ^a ($10^{15} \text{ A m}^{-2} \text{ sr}^{-1} \text{ Torr}^{-1}$)	3.0	2.2	2.8	13.0

^aNormalized to gas pressure (Torr).

The existence of an optimum temperature for GFIS was also found by Kalbitzer and Kroblasch.¹⁷ The optimum temperature is determined by the competition between the arriving rate and the escape rate of incoming gas molecules to the tip cap. The saturation ion current is mainly determined by the gas supply of the molecules, which diffuse from the tip shank to the tip apex. Increasing temperature can increase the diffusion rate, resulting in an increase of the gas supply and the ion current. However, when the temperature is increased above a certain value, the escape rate increases, so that the gas supply and the ion current decrease. Previous studies showed that the optimum temperature is close to the condensation point of the gas, which is consistent with our present measurements.¹⁷ Clearly, to operate a GFIS at the highest brightness, one should also control the tip at the optimum temperature, which varies with the gases.

The brightness and the angular intensity of four different ion beams emitted from an Ir/W SAT are calculated based on our measurements, as summarized in Table I. Because the saturation ion current increases linearly with the gas pressure,¹⁸ the brightness and the angular intensity are normalized with the gas pressure. Hydrogen ion beam is the brightest with a normalized brightness $\sim 13 \times 10^{15} \text{ A}/(\text{m}^2 \text{ sr Torr})$. Even for the least bright Ar beam, the brightness is $2 \times 10^{11} \text{ A}/(\text{m}^2 \text{ sr})$ at gas pressure of $1 \times 10^{-4} \text{ Torr}$,¹⁹ which is two orders of magnitude higher than that of Ga-LMIS. We note that the brightness can be further increased with a higher gas pressure near the tip, which can be achieved through differential pumping.⁶

To conclude, our measurements show that the Ir/W(111) single-atom tip can emit high-brightness rare gas (He⁺, Ar⁺) and reactive gas (H₂⁺, O₂⁺) ion beams with high current stability. Most importantly, this tip possesses several other good properties, including simple and reliable preparation

process, a thermally and chemically stable structure, long lifetime, and easy alignment in ion optics. All these features indicate that this single-atom tip can be a very promising candidate for future application in the GFIS-FIB systems, which will open up many new applications in nano-technology.

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¹⁹As we have confirmed the single atom sharpness, the virtual source diameter is presumably 0.3 nm.