基于碳纳米管电子源的高稳定电离真空计的性能研究

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Performance Investigation of a Highly Stable Ionization Gauge Based on Carbon Nanotube Electron Source

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Abstract In this paper, a carbon nanotube electron source is applied to a highly stable ionization gauge, and a preliminary performance experimental study of the prototype is carried out. The results show that the sensitivity of the novel ionization gauge fluctuates in the range of 0.200 Pa^{-1} ~ 0.245 Pa^{-1} from 6.87×10^{-6} Pa to 6.45×10^{-5} Pa, with a maximum deviation of 20% from the simulated sensitivity of 0.250 Pa^{-1}, and the sensitivity fluctuation is less than 5.96%, which proves that the structure is well-designed and is expected to be applied to high vacuum calibration and precise measurement.

Keywords Carbon nanotube electron source，Ionization gauge，Sensitivity

摘要 文章将碳纳米管电子源应用于高稳定电离真空计,通过结构和电参数优化,研制了样机,并开展了样机计量特性的实验研究,研究结果表明,新型电离规在 6.87×10^{-6} Pa ~ 6.45×10^{-5} Pa 的压力范围内,灵敏度在 0.200 Pa^{-1} ~ 0.245 Pa^{-1} 的范围内波动,与仿真灵敏度 0.250 Pa^{-1} 的偏差最大为 20%,灵敏度波动小于 5.96%,证明该结构设计合理,有望应用于超高真空校准及精确测量领域。

关键词 碳纳米管电子源 电离真空计 灵敏度
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1 Introduction

The ionization gauge is a device for measuring vacuum pressure. It is used to obtain the pressure indirectly by measuring the positive ion current generated by electrons colliding with gas molecules[1]. At present, the commercial cold cathode ionization gauges have a lower measurement limit of 10^{-9} Pa and the hot cathode ones have a lower measurement limit of 10^{-12} Pa. In 2021, PTB proposed a novel design to develop a high-precision ionization gauge[2], where the electron trajectories in the ionization region are fixed and only pass through the ionization region once. The emitted electrons pass through the ionization region and are collected by the Faraday cup at the end, which does not produce secondary electrons, and the electron transmission efficiency is 100%. All the ions in the ionization region are collected, and the ion collection efficiency is 100%. Performance investigation of this ionization gauge prototype show that the gauge exhibits good linearity over the measurement range of 10^{-6} to 10^{-2} Pa, with sensitivity fluctuations of ±0.5%[3]. Considering that the hot cathode has the high power consumption and thermal irradiation[4]. The carbon nanotubes (CNT) are regarded as ideal field emission cathode materials due to their high aspect ratio, mechanical strength, and low thermal disturbance[5]. Therefore, we combine a CNT electron source with the novel measurement electrodes optimized based on the basic structure proposed

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by Karl Jousten et al. The specific simulation design is described in reference [6]. The simulated sensitivity is 0.250 Pa^{-1} when the background gas is nitrogen, which is expected to further enhance the measurement performance of the highly stable ionization gauge.

2 Experimental

The highly stable ionization gauge achieves stable control of electron paths and kinetic energies by means of an electric field formed by several lens. In order to ensure the stability of the sensitivity, the emission characteristics of the electron source are crucial, so it is first necessary to carry out the morphological observation as well as the experimental tests of diode-type structure, triode-type structure and short-term emission stability of the prepared CNT cathode.

The CNT electron source was prepared by Wenzhou University using thermal chemical vapor deposition on a stainless steel substrate with the diameter of 1 mm, and its specific preparation process was described in the literature[7]. Sigma 500 high-resolution and high-precision scanning electron microscope was utilized to observe the surface morphology of CNT. As can be seen in Fig.1, the nanotubes prepared were entangled with each other, oriented randomly, with the diameter of about 40 nm ~ 60 nm, and the length of about a few tens of micrometers. The black dot present in the Fig.1(a) is due to the fact that a set of experiments had already been done on this CNT electron source prior to morphology analyzation. Normally, the black dot means the loss of the CNT caused by the joule heating or the ion bombardment.

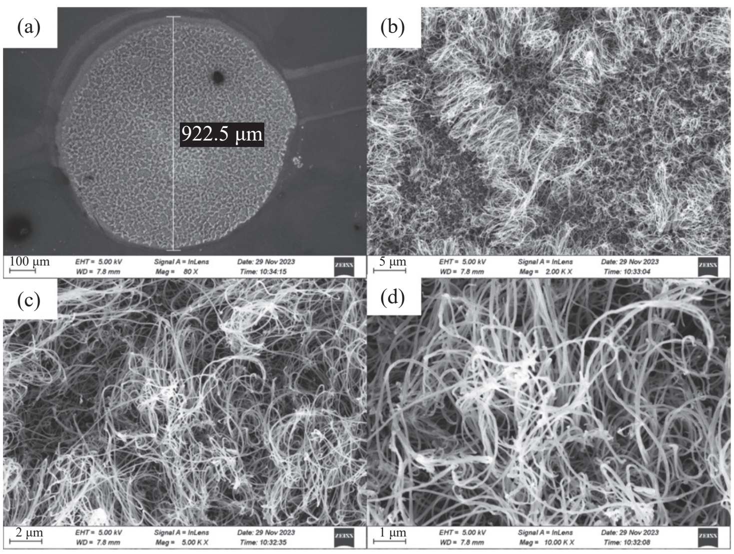

图1 碳纳米管的形貌。(a) 100 μm 尺寸的 CNT 扫描电镜图像, (b) 5 μm 尺寸的 CNT 扫描电镜图像, (c) 2μm 尺寸的 CNT 扫描电镜图像, (d)1 μm 尺寸的 CNT 扫描电镜图像

Fig. 1 Morphology of carbon nanotubes. (a) The SEM image of the CNTs at 100 μm scale, (b) the SEM image of the CNTs at 5 μm scale, (c) the SEM image of the CNTs at 2 μm scale, (d) the SEM image of the CNTs at 1 μm scale

The CNT cathode was fixed on the base when assembling the compact structure of the gauge prototype, and the tungsten mesh with wire diameter of 50.9 μm and 100 mesh was used as the gate. The spacing between the CNT cathode and the gate was controlled with ceramic spacers at 180 μm ~ 200 μm. The prototype and circuit diagram of the highly stable ionization gauge are shown in Fig.2. In order to prevent the circuit from damaging the CNT, the cathode was grounded after connecting a resistor of 10 kΩ in series.

In testing the sensitivity of the ionization gauge, an UHV/XHV calibration system[8] was used, with the background pressure in the level of 10^{-7} Pa. The standard pressure was provided using the dynamic flow method. The nine pressure points were taken to record the individual electrodes currents in the level of 10^{-6} Pa ~ 10^{-5} Pa, and the sensitivity values under different pressure points were obtained by the sensitivity calculation formula (1)[9]. Here, ΔI_+ is the change of the ion current I_+ at the ion collector before and after the introduction of the experimental gas into the calibration chamber, and I_- is the collected electron current, and the p_{std} is the standard pressure.

$$S = \frac{\Delta I_+}{I_- p_{std}} \tag{1}$$

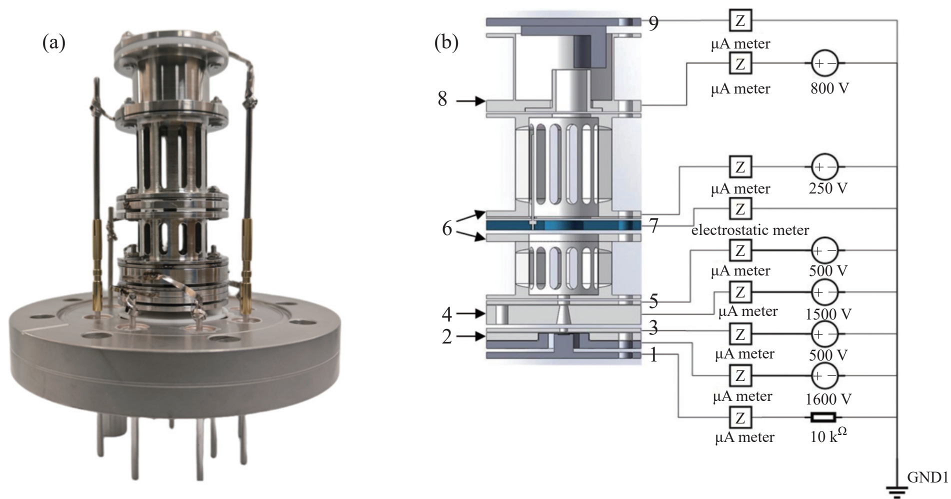

图2 高稳定电离真空规样机和电路图。(a)样机,(b)电路图(1: CNT 阴极 2: 门极 3: 减速极 4: 聚焦极 5: 入口栅 6: 电离室 7: 离子收集极 8: 电子收集极 9: 偏转极)

Fig. 2 Prototype and circuit diagram of the highly stable ionization gauge. (a) Prototype, (b) circuit diagram (1: CNT cathode 2:gate 3: deceleration electrode 4: focusing electrode 5: inlet frame 6: ionization chamber 7: ion collector 8: electron collector 9: deflection electrode)

3 Results and discuss

In order to understand the emission properties of CNT cathode, the prepared CNT cathode was first tested in diode-type and triode-type experiments. Fig.3 demonstrates to the emission properties and stability results for CNT cathode. The diode-type experimental set-up was consisted of the CNT cathode and anode, in which the anode received electrons with a stainless steel disc, and the spacing between the two electrodes was controlled by ceramic spacer at 200 μm. The anode voltage was adjusted under a pressure of 1.99×10^{-5} Pa. The cathode emission current, the anode current and their fitting curves are recorded in Fig.3(a). When the potential difference between the cathode and the anode reaches 400 V, the CNT cathode starts to emit electron current of 0.1 μA, and while the anode voltage increases to 1000 V, cathode emission current reaches 128.8 μA. Basically all of the emission current is received by the anode. The fitting curves of the cathode and the anode currents are in good agreement. The experiments are repeated to plot the *J-E* curve of the emission current density versus the applied electric field, as well as the F-N curves (Fig.3(b)) As seen from the figure, the turn-on field strength of the carbon nanotubes used in the experiments is 2 V/μm, and the emission current density reaches 15.06 mA/cm^2 when the field strength is increased to 4.85 V/μm. The field enhancement factor is calculated to be 10362.77, based on the Fowler-Nordheim (F-N) theory[10] combined with the data from the two sets of experiments.

In the triode-type experimental test, the cathode voltage was set to 0 V, the anode voltage was set to 1520 V, and the gate voltage was adjusted in the range of 0 to 1500 V. The test was carried out at a pressure of 2×10^{-5} Pa. The results obtained are shown in Fig.3(c), where the CNT cathode starts to emit electrons when the gate voltage is set at 600 V, and the anode current could be obtained when the gate voltage is higher than

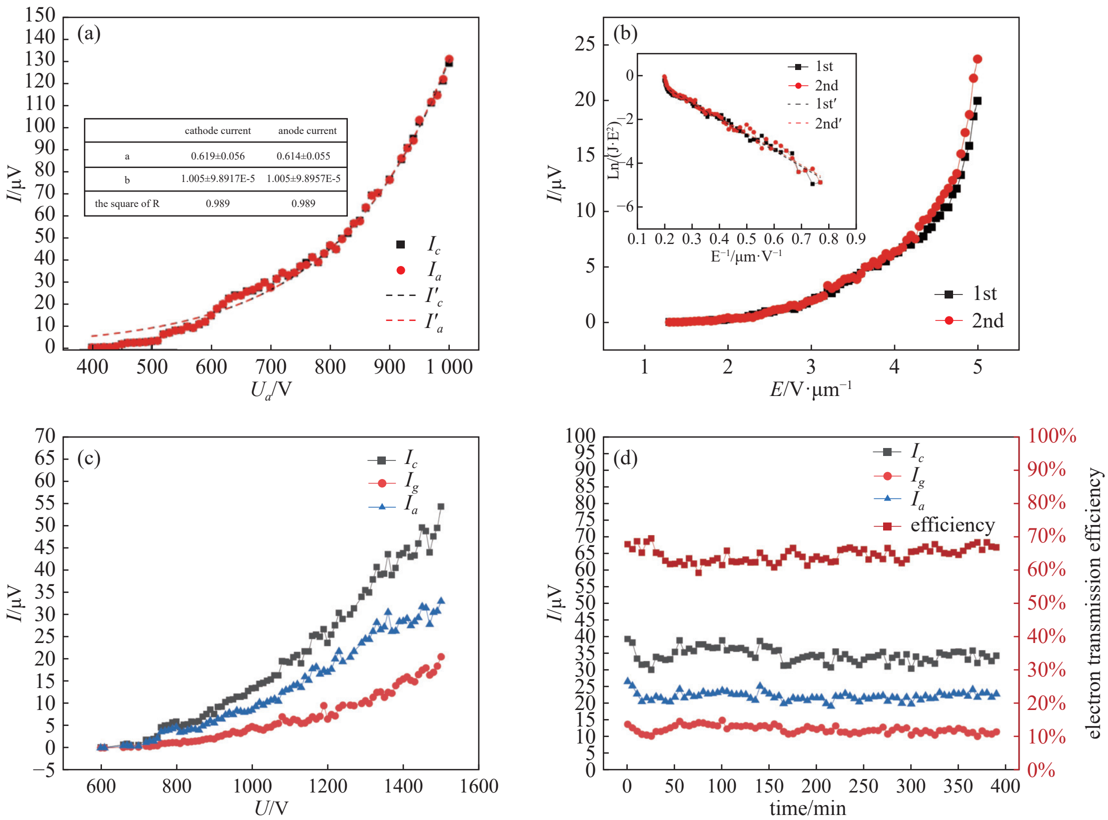

图3　碳纳米管阴极的发射特性和稳定性结果。(a)二极式结构的 *I-V* 特性曲线,(b)二极式结构的 *J-E* 特性曲线和 F-N 曲线,(c)三极式结构中阴极电流、门极电流和阳极电流随门极电压的变化曲线,(d)三极式结构中场发射阴极的发射稳定性测试(I_c 代表碳纳米管阴极的发射电流,I_g 代表门极电流,I_a 代表阳极电流)

Fig. 3　The emission properties and stability results of the CNT cathode. (a) *I-V* characteristic curve of diode-type structure, (b) *J-E* characteristic curve and F-N curve of diode-type structure, (c) curves of cathode current, gate current and anode current with gate voltage in triode-type structure, (d) emission stability tests of field emission cathodes in triode-type structures (I_c represents the current at the cathode of the carbon nanotube, I_g represents the gate current, and I_a represents the anode current)

620 V. The cathode emission current and anode current gradually increases along with the rise of the gate voltage. The emission current and anode current could reach 54.3 μA and 33 μA with the gate voltage of 1500 V, respectively.

The short-term stability of CNT emission was also investigated. The cathode emission current, gate current and anode current were tested over a period of 390 mins without stable current circuit. During the test, the cathode voltage, gate voltage and anode voltage were fixed at 0 V, 1400 V and 1500 V, respectively. The test vacuum pressure was 1×10^{-5} Pa. The results obtained are shown in Fig.3(d), where the emission current is in the range of 30.3 μA ~ 39.2 μA, the anode current ranges from 19.1 μA to 26.5 μA, and the electron transmission efficiency is maintained between 61.6% and 69.3%. It showed that CNT have stable emission and can be used as an electron source.

The triode-type electron source mentioned above was applied to the experimental testing of the gauge prototype. Based on the electrodes voltages optimization[6], the voltages of the cathode, gate, deceleration electrode, focusing electrode, inlet frame, ionization chamber, ions collector, electrons collector and deflection electrode were set as 0, 1600, 500, 1500, 500, 250, 0, 800, 0 V, respectively. The background pressure of the calibration chamber was 8.47×10^{-7} Pa. The gauge was calibrated in the range of 6.87×10^{-6} Pa ~ 6.45×10^{-5} Pa. As shown in Fig.4, the variation of the electron transmission efficiency and electron collection efficiency were recorded, and linearity correlation between the vacuum pressure and the normalized ion collection current was also investigated as well.

From Fig.4(a), it can be seen that the electron transmission efficiency is between 11.69% and 17.59%, and the electron collection efficiency is 100%, which is consistent with the simulation results[11].

From Fig.4(b), it can be seen that the normalized ion collection current is basically linear to the vacuum pressure, where the normalized ion current represents the ratio of the collected ion current to the collected electron current. The experimental sensitivities are also calculated for each calibration pressure point. From 6.87×10^{-6} Pa to 6.45×10^{-5} Pa, the sensitivity fluctuates in the range of 0.200 Pa^{-1} ~ 0.245 Pa^{-1}. According to formula (2), the sensitivity fluctuation is less than 5.96%.

$$\sigma=\frac{\sqrt{\frac{\sum_{i=1}^{9}(S_i-\overline{S})^2}{8}}}{\overline{S}} \tag{2}$$

Here, σ is the sensitivity fluctuation, i is the number of tests, S_i is the sensitivity of the calibration point, and $\overline{S}$ is the average experimental sensitivity.

The deviation of the experimental results with the simulated sensitivity of 0.250 Pa^{-1} is up to 20%. The variation of the sensitivity might be caused by the unstable electron transmission efficiency and the uncontrolled electron emission, which would be improved in further optimization.

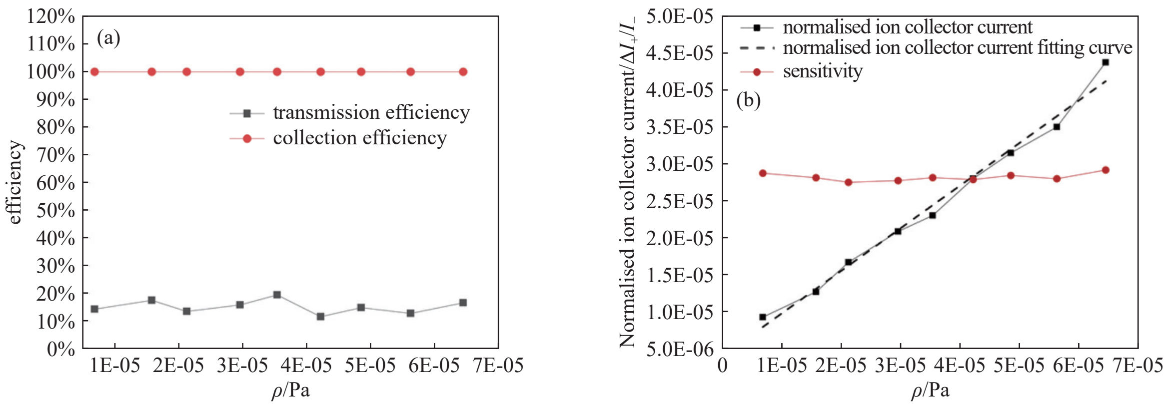

图4 整体结构的实验测试结果。(a)电子传输效率和电子收集效率,(b)归一化离子流和灵敏度

Fig. 4 Experimental test results of the overall structure. (a) Electron transmission efficiency and electron collection efficiency, (b) normalized ion collection current and sensitivity

4 Conclusion

The experimental results on the ionization gauge show that all the electrons entering the ionization chamber are received by the electron collector, which indicates that the CNT electron source applied to the highly stable ionization gauge can also realize the collimation and focusing of the electron beam. The experimental sensitivity fluctuates in the range of 0.200 Pa^{-1} ~ 0.245 Pa^{-1}, and the deviation with the simulated sensitivity of 0.250 Pa^{-1} is up to 20%, and the sensitivity fluctuation is less than 5.96%, which indicates that the design is reasonable and feasible.

However, regarding to the factors such as the emission stability of CNT, mechanical inaccuracy, and electric circuit, there will be some optimizations to be done to improve the sensitivity stability. It is expected to be applied to ultrahigh vacuum calibration or the fields requiring precise measurement.

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文章简介

目前商业化的热阴极电离真空计存在高能耗和热辐射效应等问题, 对灵敏度的稳定性会带来一定的影响。碳纳米管阴极则没有这些因素的影响, 在代替热阴极方面有巨大的潜力。本文将碳纳米管电子源应用于高稳定电离真空计, 优化了结构和电参数, 并研制了样机; 对碳纳米管电子源开展了形貌分析和发射性能测试, 并评估了样机整体结构的灵敏度稳定性。实验结果表明, 碳纳米管电子源发射的电子束得到有效准直和聚焦, 同时实现电子能量和轨迹的稳定控制, 有效避免了热灯丝的热辐射影响。实验测试的电子传输效率与仿真设计结果有较好的一致性, 高稳定电离真空计的灵敏度比较稳定, 该设计有望应用于超高真空校准和精确测量等领域。

团队介绍

研究团队长期从事真空计量及真空环境下多参数综合测试技术研究, 先后承担科技部重点研发计划、国家自然科学基金杰出青年基金、国家自然科学基金重大科研仪器等重大项目, 突破了真空中性气体、真空等离子体以及真空环境效应的系列测试计量难题, 研制了系列真空计量标准装置、电推进真空等离子体测试系统、真空测量仪器、大型真空模拟测试装置等真空装备及仪器。在国际上首次建成了常温下超高/极高真空标准装置, 率先延伸气体微流量测量下限到 10−12 Pa·m^3/s, 建立了我国较完整的真空测试计量体系。该团队先后获得“国家技术发明二等奖”、“国家科技进步二等奖”为代表的科技奖励数十项, 主导制定了真空测试计量技术领域的 ISO 国际标准、国家标准以及行业标准 30 余项。研究成果在地面、近地轨道和宇宙深空获得了全面持续应用。