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Developing Multifunctional and High Resolution *In-situ* TEM Holders

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The recent advancement of in-situ technology allows researchers have greatly expanded the application of transmission electron microscopy (TEM) from static characterization to in situ observation and dynamic measurements, providing new opportunities for understanding structure-property relation-ships of materials [1]. Commercialized *in-situ* TEM holders and platforms are mostly designed for applying a limited range of external stimuli such as heating, electrical biasing or gaseous (or liquid) flow [2]. An optical in-situ capability with a high stability becomes increasingly needed for studying the dynamic behavior of functional photovoltaic, optical-electronic or energy materials at atomic resolution. In addition, to investigate an atomic-scale structure along specific zone axes during dynamic manipulation, the double-tilt capability is also essential to be integrated in an in situ TEM holder.

In this work, we firstly designed and fabricated a multifunctional MEMS chip with both optical and electrical capabilities, which is compatible to a commercial *in situ* MEMS-based heating double-tilt TEM holder. Our in situ optical–electrical chip was also designed to adapt these four electrodes, thereby providing two electric channels, as illustrated in Fig. 1a. Because of the limited space on the chip, a side-emitting micro-LED with small size (millimeter scale) but high luminous efficiency, high response speed, long lifetime, and low cost was used a source of light illumination as shown in Fig. 1 b and c. As shown in Fig. 1d, two gold electrodes areplaced along both sides of the holes to allow the samples to be welded and thinned by FIB. For a forward current of 5mA for the blue LED (470-nm working wavelength), the maximum incident light intensity in the sample area can be as high as 150 W/m^2, which is sufficiently large to induce photon-induced current phenomena or photocatalytic reactions. Because of the low Joule heat transferred to the chip induced by the LED emission, this level of stability meets the requirements for atomic resolution imaging and spectroscopy. This provides a possible solution for function extension on commercial *in situ* platforms [3].

Furthermore, to overcome the limited probe motion as one of the major constraints on this kind of MEMS-based *in situ* holders, we will show a unique design of seal-bearing components realizing ultra-high stability and multifunctionality (including double tilting) in an in situ TEM holder. This unique seal-bearing subsystem provides superior vibration damping and electrical insulation while still maintaining excellent vacuum sealing and small form factor. This design is compatible with either a tungsten STM probing system (Fig. 2), or an optical fiber system, enabling a wide variety of in situ TEM applications including electrical measurement, STM mapping, photovoltaic studies, and CL spectroscopy with high spatial resolution imaging and electrical sensitivity at the pA scale [4]. These improvements and unique design are helpful to the future development of in situ TEM setups, and may broaden the application of in situ electron microscopy, especially for the imaging and measurements with high special resolution and precision [5].

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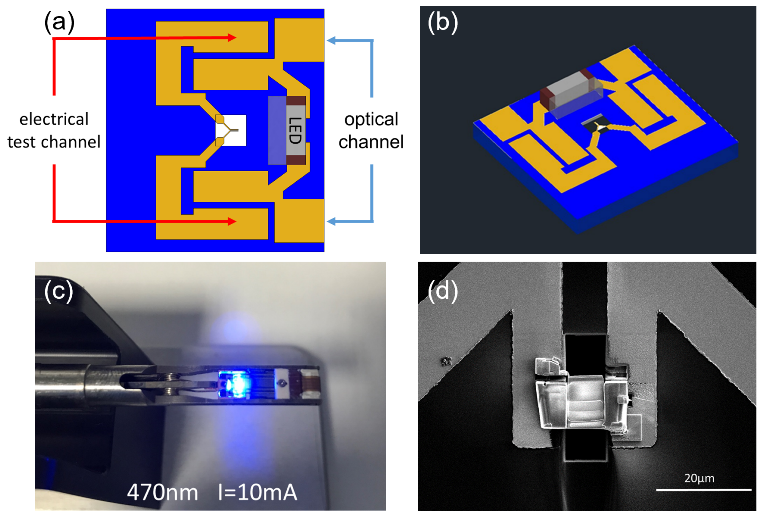

Figure 1. (a) Top and (b) perspective views of 3D model of MEMS chip. The yellow area represents the four gold electrodes on the chip surface, and the LED is connected to the middle two electrodes, facing the sample area. The outer two electrodes are connected to two micro-electrodes in the sample area. (c) This chip is carried by DENSsolution DH30 in-situ heating holder and driven by Keithley 2612B double channel SMU. (d) FIB prepared cross-section sample can be welded onto micro-electrodes for in-situ optical-electrical tests.

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