High-pressure synergetic measurement station

In the High-pressure synergetic measurement station (HP-SymS) of the Synergic Extreme Condition User Facility (SECUF), we developed ultrahigh-pressure devices based on diamond-anvil cell (DAC) techniques, with a target pressure up to 300 GPa. With the use of cryostat and magnet, we can reach 300 GPa–2.6 K–9 T and conduct simultaneous measurements of the electrical-transport property and Raman/Brillouin spectroscopy. With resistance heating and laser heating, we can reach temperatures of at least 1500 and 5000 K, respectively, coupled with Raman/Brillouin spectroscopy measurements. Some designs of supporting devices, such as a femtosecond laser gasket-drilling device, electrode-deposition device, and the gas-loading device, are also constructed in HP-SymS.

Class 100 clean room

Experimental Hall

The measurements station is mainly composed of (I) sample environment module (II) core measurement module and (III) high-pressure experiment supporting module. The specific parameters and functions are as follows:

(I) Sample environment module

a) Ultrahigh-pressure device (DAC)

The symmetrical steel, BeCu and NiCrAl DACs with different types are provided to ensure various ultrahigh-pressure experiments can be carried out above 300 GPa.

b) Low temperature–high magnetic field device

The He-free low-vibration cryostat can provide a 2.6 K-9 T environment for the DAC chamber and synchronize the *in situ* measurement of spectroscopy and electrical transport, also it is equipped with an *in situ* gas film compression device.

c) Laser heating devices

The high-power 1064 nm fiber laser can provide a 300 K-5000 K *in situ* heating environment for the DAC samples. The double-sided laser heating and temperature measurement paths can be equipped with a gas film compression device and integrated with *in situ* measurements of Raman, Brillouin spectroscopy and electrical transport.

d) Resistant heating devices

Vacuum protection is adopted, and the high-pressure experiment is carried out in a non-thermal expansion steel DAC. The temperature can be heated to 1500 K and the pressure change is less than 2 GPa.

(II) Core measurement module

a) Electrical transport properties measurements

The electrical transport properties measurements under ultrahigh-pressure mainly include DC resistance (conductivity), Hall effect and AC electrochemical impedance spectroscopy measurements. (1) The pressure dependencies of DC resistance (conductivity) and I-V characteristics of materials under high-pressure are measured by the DC current/voltage source, digital multimeter and megger. The *in situ* resistance (conductivity) and I-V characteristics measurements adopt standard four-probe or van der Pauw electrode configuration. (2) The carrier concentration, carrier mobility and Hall coefficient of materials under ultra-high pressure are measured by Hall measuring instrument with a strictly symmetrical van der Pauw electrode configuration. (3) The dielectric properties and the electric conduction in grain bulk and grain boundary of materials under high pressure are measured by AC impedance spectroscopy with a double plate electrode model.

b) Raman spectroscopy

Multi-wavelength excitation is provided, including 473 nm, 532 nm and 647 nm for *in situ* low-temperature and magnetic measurement, and 325 nm, 355 nm, 457 nm and 785 nm for *in situ* heating device, basically covering the range of visible light. The low wave number measurement can reach 10 cm^{-1}. The Raman spectroscopy can be synchronized with the *in situ* electrical measurement, and also equipped with a gas film compression device.

c) Brillouin spectroscopy

The Brillouin scattering spectroscopy system provides a 50° symmetrical scattering configuration, and can be combined with the high-temperature device or the He-free low-vibration cryostat to conduct *in situ* high/low-temperature high-pressure Brillouin spectrum measurement (the lowest temperature can reach 10 K), also it can be equipped with a gas film compression device.

(III) High-pressure experiment supporting module.

a) Electrode-deposition device

The metal electrode deposition device mainly uses magnetron sputtering to deposit metallic thin-film electrodes on the DAC to prepare the microcircuits for the electrical transport measurements of materials under ultra-high pressure. Compared with the traditional manual wiring method, the metallic film electrode is thinner and the electrode spacing is smaller, which is convenient for testing the small samples.

b) FIB/SEM

The FIB/SEM is a high-resolution scanning electron microscope/focused double ion beam microscope system. FIB can process many kinds of materials such as cemented carbide, steel and diamond. Compared with Ga ion beam, the processing efficiency of Xe ion beam is greatly improved, its acceleration to diamond is higher than 1 μm^3/min. The processing process will not introduce ion impurities, which is particularly suitable for processing the special-shaped diamond anvil used for ultra-high pressure experiment. Based on this system, we can perform micro-machining on the diamond anvils and carve the chamfer or small culet used for ultra-high pressure experiment with a precision of less than 10 nm. We can also process nano diamond (usually in micron scale) and embed it on the surface of single crystal diamond to achieve ultra-high pressure. In addition, the sample can also be processed by this machine for further characterization, such as TEM. Here, the key technology is to synchronously realize the sample processing, observation and transfer.

c) Gas-loading device

Load different gases into DAC, including H_2, CH_4, N_2, He, Ne, Ar etc., and the maximum output gas pressure reaches 200 MPa.

d) Gas-compression device

The *in situ* compression of DAC in cryogenic chamber is realized by using high pressure He.

e) Femtosecond laser drilling device

The pulse time of femtosecond laser is far less than the lattice heat conduction time. When the femtosecond laser interacts with the matter, its energy absorption is strictly limited to a small range. As a result, the electron temperature reaches extremely high in a very short time, the matter changes from a solid state to a plasma state and quickly separates from the processing body in a spray form, while the surrounding matter is still in a "cold state". Therefore, compared with traditional laser processing, femtosecond laser processing is very neat and accurate, overcoming all the drawbacks caused by thermal effects. In addition, the femtosecond processing can realize the micro processing for a variety of materials, such as metals, non-metals, semiconductors, and even transparent materials.

f) X-ray Diffraction Device for simulating second generation light source

The device adopts an Ag target with high-intensity micro-focus, the wavelength is 0.57 Å and the stability is within 0.01% fluctuation. The angle measurement device adopts a four-axis angle measuring instrument and is equipped with a high-sensitivity two-dimensional detector, which can achieve a 260 mm×260 mm test area. It also meets the focusing requirements of high-pressure micro samples and the focus size can reach 0.07 mm×0.07 mm. The X-ray diffraction experiments require high safeguard procedures, so the device is equipped with the following safety alarm facilities to protect the safety of experimental personnel: abnormal flow and pressure alarm of cooling water, abnormal generator overload detection, abnormal voltage detection, overload detection, emergency stop switch, leakage current circuit breaker, failsafe mechanism, alarm display, automatic aging function. The leakage X-ray amount of protective cover is < 2.5 μSv/h。

g) Ruby pressure calibration device

The Ruby pressure calibration device is widely used to measure the pressure in DAC. The device adopts commercial Horiba spectrometer and detector, equipped with 647 nm laser and 455 nm LED as excitation light source, which can satisfy the pressure calibration of conventional high-pressure experiment. The test results show that the device can clearly characterize the Raman signal of diamond when calibrating the DAC with pressure of 300 GPa.

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