A 1000 kV TEM Running Over 25 Years

Gerhard Kästner and Ulrich Messerschmidt

Max Planck Institute of Microstructure Physics, Halle (Saale)

A JEOL 1000 kV High-Voltage Electron Microscope has been running since 1971 when it was installed in Halle (Saale),Germany, with 280 thousand micrographs being exposed up to now .

1. Background

Halle (Saale) is an old university town located in the former East Germany (German Democratic Republic, GDR). It was in the late 60s when economy was rapidly growing even in a socialist country like the GDR. Governmental research funds were provided to expand the facilities and staff of the Academy of Sciences of the GDR. An institute belonging to this academy, the Institute of Solid State Physics and Electron Microscopy, had been established in Halle (Saale) in 1959 by Heinz Bethge. Owing to his activity and the scientific reputation of the institute, it was possible to raise the necessary amount of money for buying a JEOL high-voltage transmission electron microscope (HVEM), serial # 6, operated at 1000 kV. Worldwide, there were about 30 HVEMs running in the 70s — mainly in Japan, the UK, and the U.S.A., among them two in Stuttgart/Germany and one each in Stockholm/Sweden, Moscow/USSR and Halle (Saale) /GDR.

The HVEM in Halle (Saale) — shown in **Fig. 1** — was installed in a new dedicated building between 1970 and 1971. From the very beginning of constructional work, a staff of a leading scientist (one of the authors, G. K.), two technicians, and an increasing number of scientific co-workers were involved in all details. It was very stimulating to assist the Japanese team of up to five engineers in the assembly and during the test period of all parts of the big machine. Various provisional installations had to be made, including a wooden 1 m^2 basement for a heavy (1000 kg) motor-generator supplying the 3 kHz input of the high-voltage system. When connecting this provisional installation for the first time, all persons kept at a safe distance of several meters. Right from the beginning, the staff was involved in all details of the later operation, including the scheduled annual overhaul, according to a five-year contract with JEOL. Subsequently, the staff was able to overhaul the whole system — including the complete disassembly and cleaning of the high-voltage system and the accelerator tube — with only occasional help by JEOL. This was essential as it saved much money in convertible currency, which was very rare in the GDR. Later on, it turned out that the accelerator was working well for several years without cleaning, and the annual downtime for repair and cleaning other parts was reduced to about 3 weeks. Up to now, more than 280,000 micrographs have been exposed.

In general, it was advantageous to have an experienced team of permanent employees. Thus, a lot of difficulties could be solved which arose from the chronic shortage of materials and convertible currency typical of all socialist economies. Two examples may be mentioned: After running the microscope for about 6 years, a set of 20 spare insulators for the 1 MV accelerating vacuum tube was managed to be produced in a GDR factory according to drawings kindly provided by JEOL. It was a similar but more tedious in-house work to design and build a new 1 MV resistor column — located within the high-voltage gas-pressurized tank — as the measuring resistor to provide the feedback voltage for the 1 MV voltage control and stabilization circuit. After several trials, the 14 stages of this column were built from 4480 thin-film resistors individually pre-tested. This resistor column has been working well for 4 years.

The background changed drastically after the reunification of Germany in 1990. Since 1992, the HVEM facility has been part of the new Max Planck Institute of Microstructure Physics established in Halle with the directors of the experimental departments Ulrich Gösele and Jürgen Kirschner. The instrument has now mainly been used for *in situ* investigations. Accordingly, the responsibility has been passed to the other author (U. M.). Owing to the skill of Christian Dietzsch (physicist) and Wolfgang Greie (technician), both from the most experienced former staff, all technical problems and breakdowns have been solved without help from outside so that the microscope is still working stably most of the time. Because of the still high demand for conventional applications and, particularly, because of the unique possibilities of *in situ* experiments, it is planned to maintain the instrument also over the next few years.

2. Technical upgrade and *in situ* facilities

Because of the low budget of convertible currency available during GDR times, the HVEM was bought at a very low price level so that its technical facilities had to be upgraded and expanded in various ways. Nearly all improvements were laboratory-made in order to achieve unique facilities.

2.1. Upgrade

At the very beginning, two upgrades badly needed were done: a film numbering device (where an electromechanical display is optically exposed) and an automatic starting unit, switching on the pumps, etc. after sudden power cuts (which were quite often during construction work within the campus). This starting unit was built by using electromechanically driven switches since no IC´s were available. In addition, the noisy 3 kHz transformers of the high-voltage supply were put in insulating boxes (and therefore water-cooled) to ensure sound protection.

Later on a TV system was installed, consisting of a transmission fluorescent screen, a 45° mirror, a lens-coupled TV camera, and an extension of the X-ray lead shielding.

A quite useful feature of the HVEM is the large diameter (about 300 mm) of its specimen chamber as well as the diameter of 36 mm of the upper bore of the top-entry objective pole piece. This provides enough room for attaching special cartridges and devices.

A precise universal goniometer was built in 1977 by one of the authors (G. K.) in order to improve and expand the routine facilities for using electron diffraction and diffraction contrast. First, the specimen airlock and top-entry transfer were completely redesigned to better manipulate à new standard specimen cartridge for larger samples. It may clamp specimens of up to 8 mm in diameter and 1 mm in height. This cartridge also enables double-tilting of up to 45° at all azimuth angles by means of the top-entry goniometer mechanism shown in **Fig. 2**. The cone-shaped specimen cartridge C (shown separately on the right) is kept by a cartridge holder H, which is supported by a universal joint precisely located within the specimen plane of the objective lens. At about 13 mm above this plane, a small brass ball is fixed to the cartridge holder (below the letter H). This ball is gripped between actuating tongs T, each arm coupled to a horizontal gear drive G. On moving these drives into opposite directions, the opening angle of the tongs is varied, thus tilting the cartridge in y direction. If the drives are moved synchroneously into the same direction, the tongs translates without altering the angle, thus tilting the cartridge in x direction. Tilt angle and azimuth are displayed in two dimensions on a screen in front of the operator. This screen is shown as a bright area above the viewing chamber in Fig. 1. It is connected to the vertical tilting drive rods with their handle knobs conveniently located on the left and right sides of the viewing window.

2.2. *In-situ* facilities

Together with the microscope, various top-entry attachments for *in situ* investigations were bought:

Specimen heating stage:
An airlock-exchangeable specimen cartridge can be resistance-heated up to 1070 K and tilted up to 30° in any direction.

Specimen cooling stages:
Airlock-exchangeable specimen cartridges can be cooled by liquid N_2 down to 150 K with double-tilting facilities up to 10°, or down to 120 K without tilting.

Transversal magnetic field stage:
On inserting this stage into a specially shielded objective pole piece, a horizontal magnetic field of up to 30 kA/m can be applied across the specimen plane and rotated mechanically into all azimuths.

Tensile stage:
A specimen can be fixed to a non-tiltable stage where a blade spring extends the specimen, controlled by heating a wire.

After some years of work and respective experience, a number of new stages were laboratory-made in order to meet particular requirements and to extend the facilities.

2.2.1. Heating/tilting stage

To attain higher temperatures, a new stage was built in 1983 [1] where the tantalum/molybdenum specimen cartridge is heated by electron bombardment from a surrounding W filament, a method which was introduced into the design of *in situ* stages by Fujita and Komatsu [2]. The cartridge holder, including the filament heating and high-voltage leads as well as water cooling and a thermocouple, can be double-tilted up to 23° via the tongs mechanism shown in Fig. 2. The cartridge itself is exchangeable via the redesigned specimen airlock transfer mechanism. Temperatures of

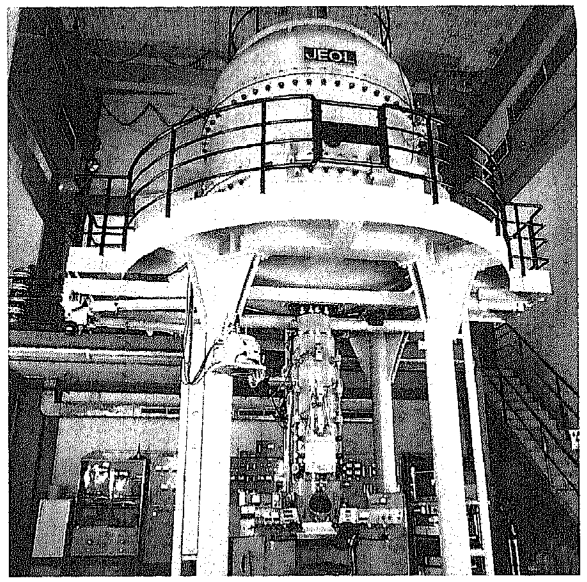

Fig.1. The JEOL 1000 kV high-voltage transmission electron microscope installed in Halle (Saale), Germany.

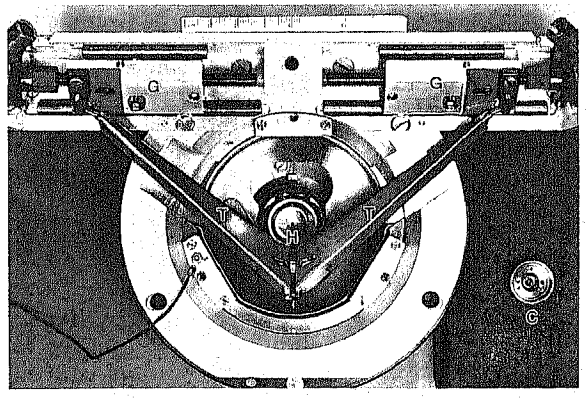

Fig.2. Laboratory-made universal 45° double-tilting stage viewed from the top. The specimen cartridge C shown separately is airlock-transferred to the cartridge holder H. It is tilted on moving the tongs T by means of gears G (G. Kästner, 1977).

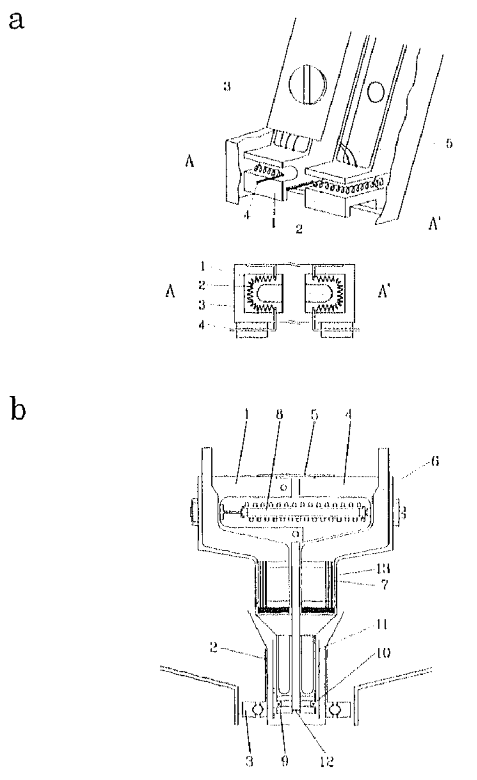

Fig.3. Schematic drawing of the double-tilting high-temperature straining stage. a) Perspective view of the hot zone. 1 - specimen grips, 2 - coil filaments, 3 - thermal shields, 4 - electric connections of filaments. b) Drive and cooling system. 1 - fixed lever, 2 - tilted cone carrying the deformation device, 3 - ring of cardanic suspension of the double-tilting stage, 4 - moveable lever, 5 - leaf spring, 6 - cooling water pipe, 7 - lamellae heat exchangers, 8 - stainless steel tube with heating coil, 9 - W-27Re specimen grips, 10 - W filaments, 11 - thermal shields, 12 - specimen, 13 - seminconducting strain gauges (U. Messerschmidt, 1994, after [4]).

Halle (Saale), D-06120, Germany
E-mail: kaestner@mpi-halle.mpg.de