Investigating the crystal structure of tungsten using a field emission microscope

Objects of the experiments

- Observing the body-centred cubic structure of a tungsten monocrystal with a field emission microscope.
- Observing individual barium atoms and their thermal motion on the surface of the tungsten monocrystal.

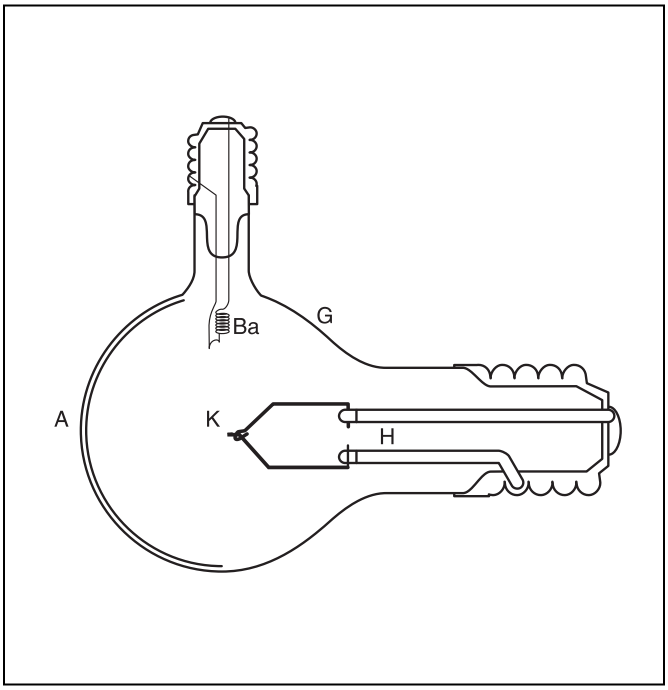

Fig. 1 Field emission microscope after E. W. Müller

Principles

In the field emission microscope, an extremely sharp tungsten tip is placed as a cathode K in the centre of a spherical glass tube G under high vacuum (see Fig. 1). A fluorescent screen is attached to the inside wall of the glass tube as an anode A. As can be observed in a darkened room, the fluorescent screen lights up as soon as there is a high voltage of at least 5 kV between the cathode and the anode because then electrons are emitted from the cold tungsten tip through field emission (tunnel effect) and reach the fluorescent screen.

The tungsten tip is the monocrystalline end of a thin wire bent into a loop. It is made in an etching procedure. At first, the tip is sharp-edged and rather irregular. By subsequent glowing in a high vacuum, it takes the shape of a hemisphere with a radius $r \approx 0.1–0.2\ \mu\text{m}$. This hemisphere can not be studied with a usual light microscope. It is almost perfect except small plane areas at crystal planes of low crystallographic index.

When a high voltage is applied, the field lines run radially from the hemisphere to the fluorescent screen. Near the hemisphere, the electric field comes up to field strengths of the order of magnitude of 10^{10} V m^{-1}; conduction electrons can, therefore, escape from the tungsten crystal through field emission and move radially towards the fluorescent screen. Field emission depends strongly on the electron work function. The work functions of different crystal planes, on the other hand, differ a lot from each other. On the fluorescent screen, an image of the structure of the tungsten tip appears magnified by the factor

$$V = \frac{R}{r} \qquad \text{(I)},$$

R = 4 cm: radius of the fluorescent screen.

The image is composed of a regular order of bright and dark spots.

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Apparatus

1 field emission microscope	554 60
1 FEM connection panel	554 605
1 pair of stand feet	301 339
1 high voltage power supply 10 kV	521 70
1 variable extra low voltage transformer . .	52139
1 amperemeter, up to 10 A . . . . e.g.	531 712
safety connection leads	

Safety notes

The field emission microscope is an interference radiator as defined by the German X-ray regulations (RöV), because at high voltages greater than 5 kV X-radiation is produced. If the tungsten tip is intact, safe operation is ensured: The dose rate remains certainly below the permissible maximum value *j* = 1 µSv/h. If the tungsten tip is defective, excessive dose rates may result under unfavourable conditions.

- Mind the instruction sheet of the field emission microscope.
- Do not operate a field emission microscope with a defective tungsten tip (luminance phenomena with sharp boundaries on the fluorescent screen) at high voltages greater than 5 kV.

The tungsten tip may be overheated locally by too high currents and, as a consequence, melt so that the field emission microscope is irreparably destroyed.

- Under no circumstances bake the tungsten tip out when high voltage is applied.
- Before baking out the tungsten tip, switch the high voltage off. While baking out the tungsten tip, watch the amperemeter (the heating current must not exceed *I* = 1,8 A).
- Before applying the high voltage after baking out, allow the tungsten tip to cool down.
- While the high voltage is applied, do not heat the tungsten tip beyond dull red heat for observing the barium atoms.

During operation, the field emission microscope and at the FEM connection panel are under high voltage. In the connection proposed, the anode and the heating for the barium supply are on earth potential. This is only possible on condition that a high-voltage proof heating voltage source is used to heat the tungsten tip.

- Use the 6.3-V output of the high voltage power supply 10 kV (521 70) as a heating voltage source.
- Operate the FEM connection panel only when the housing cover is closed.
- Establish and change connections only when the power supplies are switched off.

The field emission microscope is a high-vacuum glass tube, and thus extremely sensitive to shocks.

- Handle the field emission microscope carefully and protect it from mechanical shocks even after connecting it to the FEM connection panel.

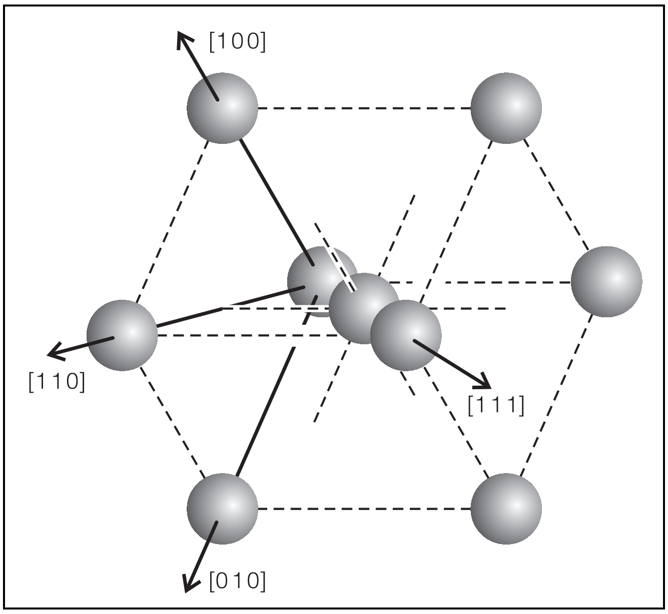
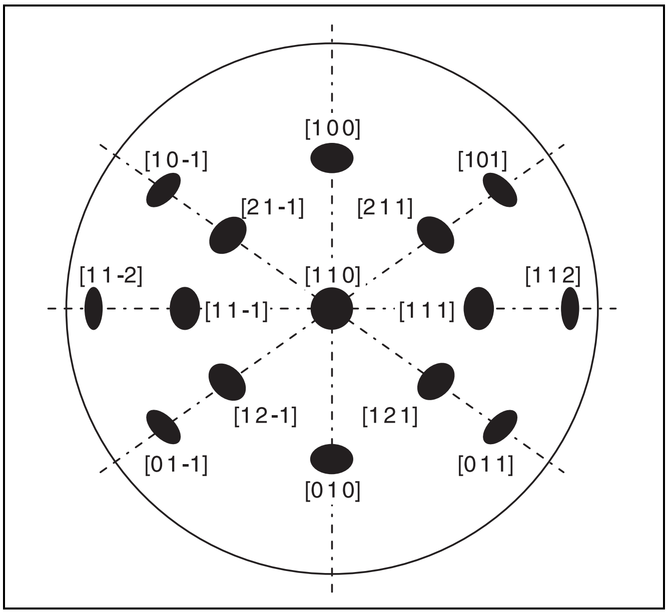


Fig. 2 Unit cell of the tungsten lattice

Imaging the structure of tungsten:

In Fig. 2, the body-centred cubic unit cell of the tungsten lattice is shown. The crystallographic direction [1 1 0] points to the centre of the fluorescent screen since it – due to the way of preparing the crystal – coincides with the longitudinal direction of the tungsten tip. The directions [1 0 0] and [0 1 0] point upwards and downwards respectively. The penetration points of these directions and some other directions of low index are shown in Fig. 3 in parallel projection. A corresponding image of the tungsten crystal can be observed on the fluorescent screen after the tungsten tip has been cleaned of foreign atoms adsorbed on the surface by heating it up to white heat. As atoms and molecules of the residual gas in the field emission microscope are adsorbed on the tungsten tip, the image on the fluorescent screen changes permanently during some minutes of observation [1–3].

Fig. 3 Indices of the directions of emission as seen on the fluorescent screen in parallel projection



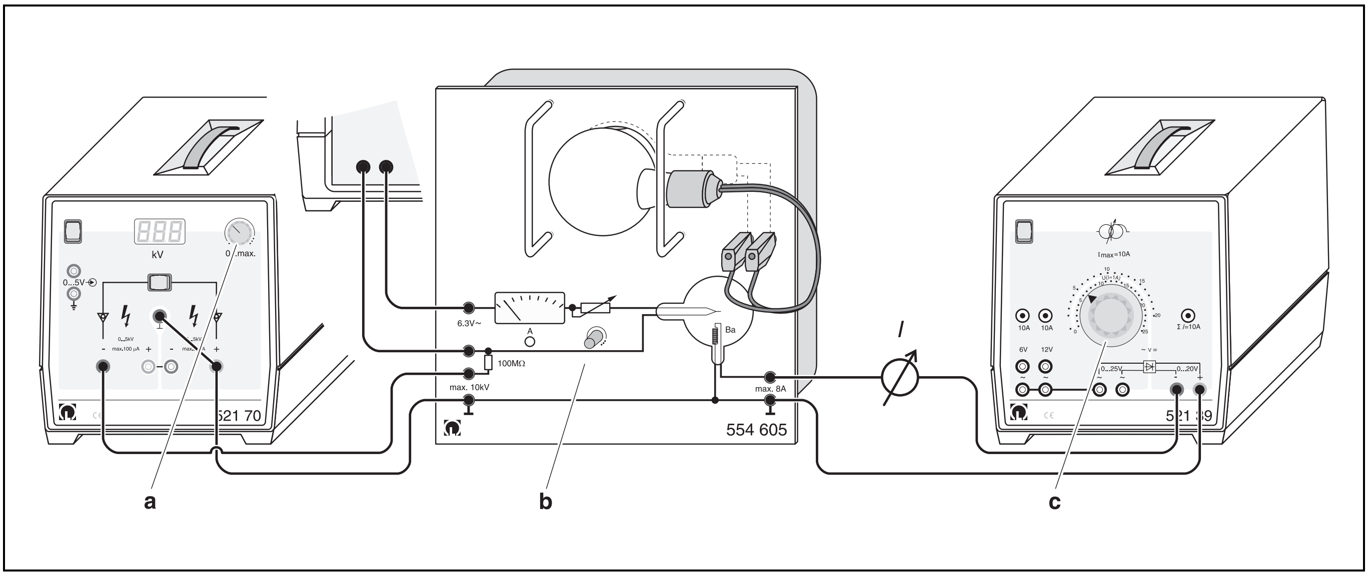


Fig. 4 Experimental setup with the field emission microscope.

Imaging barium atoms:

The residual gas atoms are too small for observing individual atoms. However, atoms of the electropositive metal barium have a diameter of about 0.4 nm and are thus big enough to be seen as bright spots on the fluorescent screen when they have condensed on the tungsten tip. These bright spots are even bigger than they would be according to Eq. (I) because the barium atoms influence the course of the field lines at the tungsten tip [4].

For the observation of vapour-deposited barium atoms the field emission microscope contains, apart from the cathode, a ring-shaped trough with a barium supply Ba (see Fig. 1). This trough is at anode potential and can be heated electrically. If a tiny portion of barium is vaporized in the glass tube, individual barium atoms will condense on the tungsten tip and cause bright spots lighting up on the fluorescent screen. If the tungsten tip is heated cautiously, the thermal motion of the barium atoms can be observed. As the atoms change their positions, the bright spots light up and fade away.

Setup

The experimental setup is illustrated in Fig. 4. The anode and the barium supply are on earth potential. Any standard power supply, e. g. the variable extra low voltage transformer (521 39), may be used to heat the barium. For baking out the tungsten tip, however, a high-voltage proof heating voltage source is required, e. g. the 6.3-V output of the high voltage power supply 10 kV (521 70).

- Mount the field emission microscope in the FEM connection panel (see instruction sheet for the FEM connection panel).
- Set the high-voltage controller **(a)** and the potentiometer **(b)** for the heating current to zero (left stop).
- Connect the high voltage output and the 6.3 V output of the high voltage power supply to the FEM connection panel as shown in Fig. 4.
- Turn the knob **(c)** of the variable extra low voltage transformer to the left stop, connect the variable extra low voltage transformer to the heating of the barium, and connect the amperemeter in series.

Carrying out the experiment

Remark: The room in which the experiment is carried out should be as dark as possible because then a lower high voltage is sufficient.

- Switch the high voltage power supply on to check the tungsten tip, slowly increase the high voltage, and observe the image on the fluorescent screen in the darkened room.

In case that luminescent phenomena with sharp boundaries suggest that the tungsten tip is defective:

- Do not operate the field emission microscope with high voltages greater than 5 kV.

Baking out the tungsten tip:

- Set the high voltage to zero, and wait until the output voltage has returned to zero.
- Starting from zero, increase the heating current slowly (!) up to the maximum value of 1.6–1.7 A, and let it flow for about 5 minutes (the tungsten tip lights up brightly).

Studying the clean tungsten tip:

- Set the heating current for the tungsten tip to zero.
- Starting from zero, increase the high voltage slowly up to about 7 kV.
- Observe the image on the fluorescent screen for some minutes.

Vapour deposition of barium:

- The high voltage being applied, increase the heating current of the barium supply slowly up to about 7.5 A or at most 8.0 A, starting from zero, and observe the image on the fluorescent screen.

After heating for some seconds, you will at first observe a slight movement in the image on the green fluorescent screen. Then clusters of bright spots will light up spontaneously around the large dark areas of the image.

- Stop heating the barium supply not later than after 30 or at most 60 s.

Observing the tungsten tip with the vapour-deposited barium:

- Increase the heating current of the tungsten tip to about 0.5 or at most 0.6 A (dull red heat), and observe the image on the fluorescent screen.

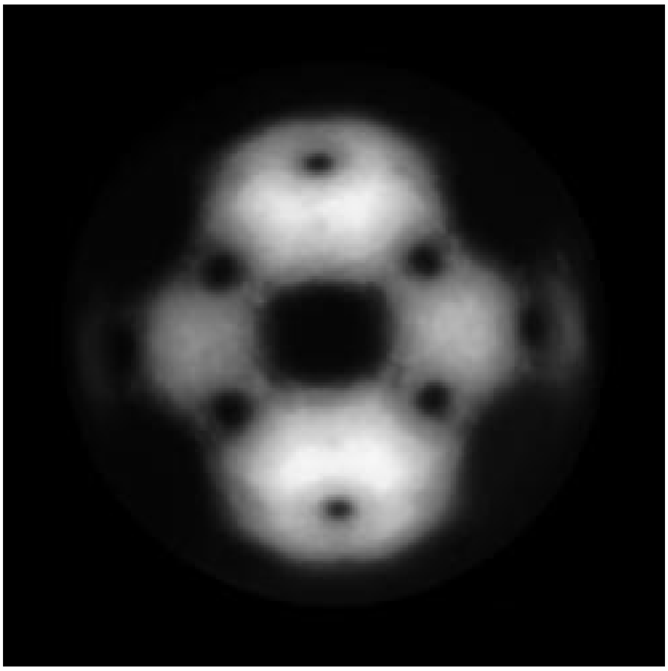


Fig. 5 Image of the clean tungsten tip immediately after baking it out.

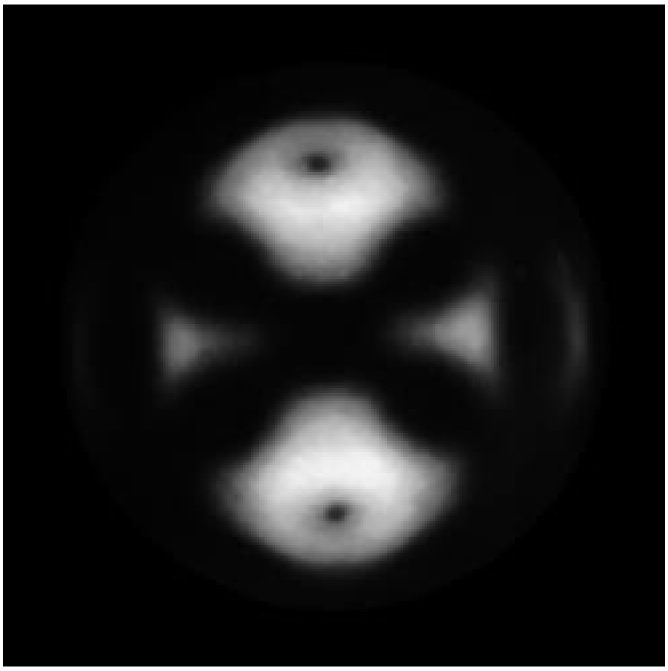


Fig. 6 Image of the clean tungsten tip about 3 minutes after the tip has been baked out.

Measuring example and evaluation

Clean tungsten tip:

Fig. 5 shows the image of the tungsten tip cleaned by heating it up to white heat. The bright and dark regions are easy to identify by comparison with Fig. 3:

The plane (1 1 0), which is perpendicular to the direction [1 1 0], is mapped onto the centre of the fluorescent screen as a large dark area. All other planes with the same crystal symmetry, that is (1 0 1), (0 1 1), (1 0–1) and (0 1–1), appear dark too. From these planes only few electrons are emitted because the work function of these planes is greater than that of the other planes.

The crystal planes (1 0 0) and (0 1 0) as well as (2 1 1), (1 2 1), (1 1 2), (2 1–1), (1 2–1) and (1 1–2) are mapped as small dark regions. They have medium work functions.

The images of the planes (1 1 1) and (1 1–1) are bright. Their work functions are relatively small.

Residual gas atoms on the tungsten tip:

After about 3 minutes, the image has already changed considerably, since the tungsten tip is covered with atoms of the residual gas (see Fig. 6).

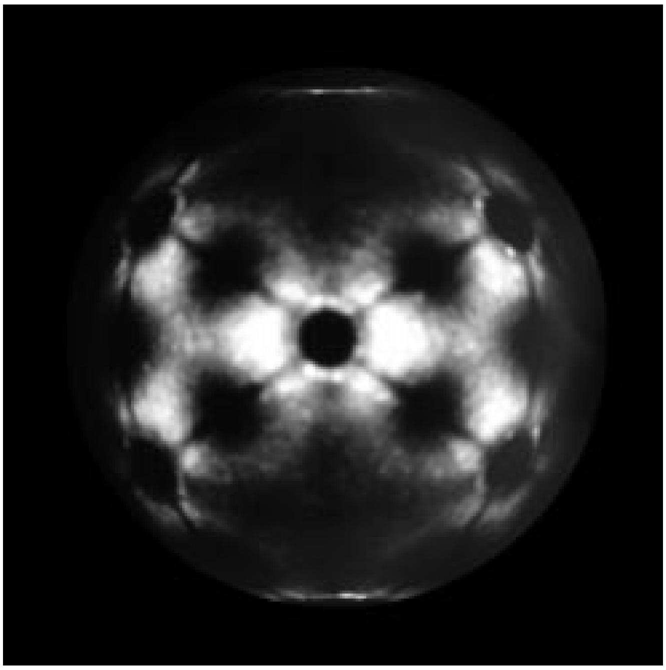
Observing the tungsten tip with the vapour-deposited barium:

After barium has been vapour-deposited on the tungsten tip, the motion of individual barium atoms can be observed if the tungsten tip is heated cautiously. Scattered bright spots indicate that barium atoms change their positions. This effect is particularly visible in the dark regions. The thermal lattice vibrations become strong enough for the barium atoms to leave their sites against the adsorption forces. With a statistical distribution single atoms are freed. The lateral motion of the barium atoms itself cannot be seen.

This process is at first observed at the {1 0 1} planes, that is, at the edge of the fluorescent screen. These planes appear more clearly than in the image of the clean tungsten tip because their boundaries are marked by bright luminescent images of barium atoms.

Later on, the barium atoms start changing their positions at other sites too. Obviously the adsorption forces are smallest on the {1 0 1} planes and greatest on the {1 0 0} planes because on the latter the motion of the luminescent spots starts latest.

Fig. 7 Image of a tungsten tip with vapour-deposited barium



If the temperature is increased a bit more, the motion of the barium atoms becomes so lively that it soon becomes impossible to identify individual barium atoms. At this stage, the images of barium atoms that are visible in the bright regions begin to move. Due to the lively motion individual barium atoms can no longer be discerned. On further increase of the temperature, the barium atoms disappear more and more from the bright regions and contract to characteristic places between the dark regions. At these locations, the motion of the barium atoms is very lively.

Supplementary information

If barium atoms are vapour-deposited on the tungsten tip while the high voltage is switched off, they condense mainly on the side facing the barium supply. Here, several layers of atoms may be deposited one on top of the other. After applying the high voltage, the image on the luminescent screen has a bright luminescent edge. This edge corresponds to the monoatomic boundary of the deposited barium.

Now the barium can be distributed over the whole tungsten tip by heating the tip. Again, start with a very low heating voltage. More and more barium atoms will be observed to move onto the uncovered part of the tungsten tip. The dark regions are largely left free because the adsorption forces acting on the barium atoms are weaker there. Finally the barium atom appear in the bright regions of the image as fluctuating spots.

Bibliography

[1]E. W. Müller, Zeitschrift für Physik, 1937, 106, 541.

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[4]E. W. Müller, Zeitschrift für Naturforschung, 1950, 5a, 473