

# The Arrangement of the Micro-crystals in the Film of Molybdenum obtained by Deposition

By

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## Abstract

By means of Laue-photographs the arrangement of the micro-crystals in the thin film of molybdenum obtained by deposition was studied. It was found that they are composed of micro-crystals which are so arranged that the (110) plane is almost in the flat surface of the film and a cube edge is nearly parallel to the lengthwise direction, but some of them are composed of somewhat large micro-crystals, which are arranged fibrously with the [110] axis as fibre axis which is normal to the flat surface of the film.

The arrangement of the micro-crystals in deposited metals has already been studied by many investigators; and especially S. Rembinska<sup>1</sup> studied the crystal structure of thin metallic films prepared by cathodic or thermal sputtering by means of a Bragg apparatus. In the present experiment the arrangement of the micro-crystals in molybdenum deposited on a molybdenum anode was examined by means of Laue-photographs.

The deposited molybdenum crystals examined were obtained by the process of heating a molybdenum anode sufficiently by electron bombardement in vacuo so that molybdenum was removed from the anode and was deposited as a thin film on the inner surface of the glass tube in which the anode and cathode were sealed; and then, owing to the increased deposition of the molybdenum on the surface of the glass tube, the deposited film flaked off from it and was deposited reversely on the anode. The apparatus used in this deposi-

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1. S. Rembinska: *Zeit. f. Phys.*, **54**, 46 (1929).

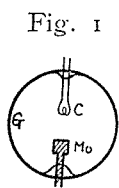


Fig. 1 tion is diagrammatically shown in Fig. 1, where Mo is the molybdenum anode, C the hot cathode of tungsten filament and G the spherical glass tube of about 10 cms. in diameter. The potential applied to the tube was about 40 K. V. s., the electronic current passed through 15-20 milliamperes, and the bombardment was carried out for about 200 hours till the deposits came to appear on the anode. Some of the molybdenum deposits thus obtained are shown in Figs. 1 and 2, Plate I, where we can see needle-shaped and dendriform crystals standing perpendicularly to the surface of the anode. In Fig. 3, Plate I, the deposited films which were flaked off from the inner surface of the glass tube, and their traces, are shown.

It was found by a microscopic examination that most of them are thin films composed of the aggregates of the micro-crystals and that some of them are composed of single crystals as shown in Figs. 4, 5, 6 & 7, Plate I, where Figs. 5 & 7 are the enlargements of some parts of the photographs shown in Figs. 4 and 6 respectively.

Then the ordinary Laue-photographs were taken with these crystals by sending a narrow and circular beam of X-rays, which started from the focus on the Mo-target of a Coolidge tube, obliquely to the flat surface of the specimen, the photographic plate being set perpendicularly to the beam of the X-rays. Some of the typical X-ray radiographs thus taken are shown in Figs. 8, 9, & 10 in Plate I, where Figs. 8 & 10 are the Laue-photographs taken with the crystal shown in Fig. 4, Plate I, and Fig. 9, that taken with the crystal shown in Fig. 6, Plate I respectively. In these cases the beam of incident X-rays was made to strike the crystal perpendicularly to its lengthwise direction and obliquely to the normal to its flat surface. In the cases of Figs. 8, 9 & 10, Plate I, the beam of X-rays was inclined to the normal at angles of about  $25^\circ$ ,  $25^\circ$  and  $10^\circ$  respectively, and in each figure an arrow indicates the lengthwise direction of the specimen.

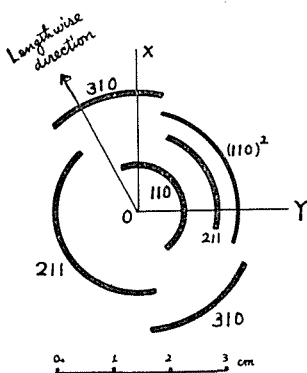
With these radiographs the orientation of the crystallographic axes was determined by treating the Laue-spots with the crystallographic globe.<sup>1</sup> It was found that though the crystal shown in Fig. 6, Plate I, seems to be a single apparently, it is composed of micro-crystals which are so arranged that the (110) planes are in the flat

1. U. Yoshida: Japanese, J. Phys., 4, 133 (1927); S. Takeyama: These Memoirs, 12, 257 (1929).

surface of the crystal and the cube edges nearly parallel to the lengthwise direction, with some scattering around the lengthwise direction. In the crystal shown in Fig. 4, Plate I, the micro-crystals are arranged fibrously with the  $[110]$  axis as their fibre axis which is normal to the flat surface.

Thus, I assumed that the  $[110]$  axis which is normal to the flat surface is the fibre axis of the micro-crystals and that the beam of incident X-rays strikes the specimen with the inclination of 10 degrees to the normal to the flat surface and perpendicularly to the lengthwise direction, and then I calculated the diagram of the diffraction pattern which might be expected to appear on the photograph. The agreement between the diagram thus calculated and the photograph shows the correctness of the conclusion given above. The calculated diagram Fig. 2, which was drawn by taking  $10^\circ$  as the angle of inclination of the beam of X-rays to the normal to the flat surface of the specimen, corresponds to Fig. 10 in Plate I.

Fig. 2.

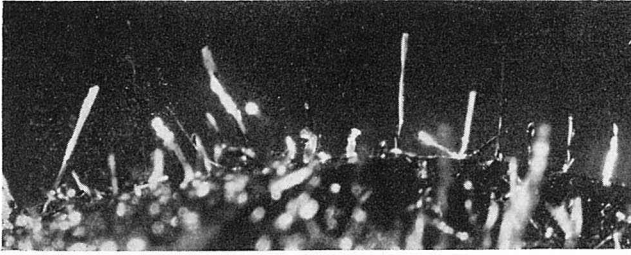


In conclusion, I wish to express my sincere thanks to Prof. U. Yoshida, of Kyoto Imperial University for the interest he has taken in this research.

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Plate I

Fig. 1



× 8

Fig. 2



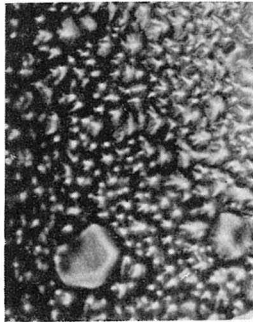
× 8

Fig. 4



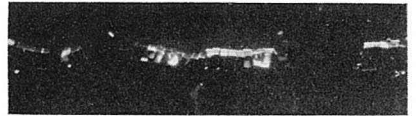
× 26

Fig. 5



× 650

Fig. 3



× 10

Fig. 7



× 650

Fig. 6



× 26

Fig. 8

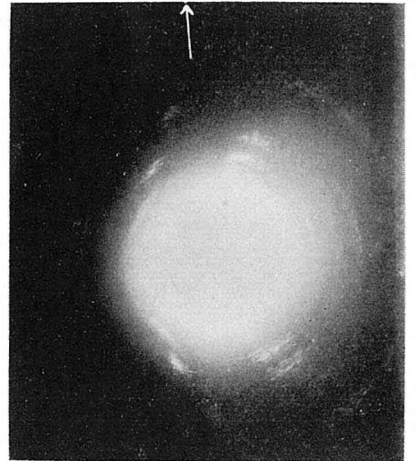


Fig. 10

Fig. 9

