

The Structure of Evaporated Films of Chromium and Aluminum on Glass

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(Received December 29, 1941)

The structure of evaporated films of aluminum on glass, and of aluminum on chromium deposited on glass, was investigated by electron diffraction. No evidence was found that would indicate the nature of the hardening produced in aluminum films by using a chromium base or by washing with water. The oxide film on aluminum, if present, is very thin and amorphous.

MIRRORS made by evaporating films of aluminum on glass are widely used in physics and astronomy. It has been claimed that such aluminum films are more resistant to scratching if deposited on an evaporated chromium film on glass than if deposited on glass directly, and also that the films are hardened if washed with water after preparation.¹ An electron diffraction study of these films was undertaken in order to determine their structure, with particular reference to the hardening effect of the chromium base and the treatment with water.

The electron diffraction camera was a modified form of the Thomson-Fraser camera,² with a hot filament electron source. The accelerating voltages were 25 to 30 kv. A plate magazine made it possible to take five photographs in succession without opening the camera. Thus the films, which were prepared in the camera, could be examined during preparation without exposing them to air.

The technique used in evaporating the films followed as closely as possible the methods used in the manufacture of aluminum mirrors. A piece of glass about 1 cm square was cleaned by washing with potassium hydroxide, hydrochloric acid, and rinsing with distilled water. When dry it was placed in the camera, care being taken to avoid contaminating it in handling. Opposite the specimen holder were electrical leads to which were fastened spirals of 15-mil tungsten wire, one containing chromium, the other containing aluminum. The camera was evacuated and after the pressure had been reduced to

5×10^{-5} mm of Hg or less, the filaments were heated by a small current to drive off impurities. The filament containing chromium was then heated by a current of 15 amperes and the chromium evaporated. The progress of the evaporation could be watched through a window in the apparatus. After the chromium was deposited a photograph of the diffraction pattern produced by the film was obtained. Aluminum was then evaporated on the chromium. Two photographs of the diffraction pattern were taken, one after a small quantity of aluminum had been evaporated, the other after a thick film was deposited. The camera was then opened and the specimen exposed to air, after which another photograph was taken to determine whether any change in the diffraction pattern could be detected after such exposure. Still another record of the diffraction pattern produced by the film was made after the film had been rinsed with water.

The diffraction pattern produced by the chromium film could be explained completely in terms of the body-centered cubic structure of chromium as determined by x-rays ($a = 2.88\text{\AA}$). In most of the films there was preferred orientation, with the (111) plane parallel to the surface of the glass. None of the films showed any trace of the presence of an oxide of chromium. Beeching,³ who has also investigated the properties of chromium films on glass, found that with thin films he obtained a pattern containing oxide rings. This may have been because the films he studied had been produced by evaporation at a higher pressure (10^{-4} mm of Hg) than in the present case.

The thick films of aluminum on chromium

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¹ G. B. Sabine, *Phys. Rev.* **55**, 1064 (1939).

² G. P. Thomson and C. G. Fraser, *Proc. Roy. Soc. A* **128**, 641 (1930).

³ R. Beeching, *Phil. Mag.* **22**, 938 (1936).

gave the pattern to be expected from the face-centered structure of aluminum. No rings were present which could be attributed to aluminum oxide. The aluminum showed preferred orientation on the chromium, with the cube face parallel to the surface of the glass. It was noted that the distance between adjacent atoms in the (111) plane of chromium ($\sqrt{2} \times 2.88 = 4.07\text{A}$) was almost the same as the distance between alternate atoms in the cube face of aluminum ($a = 4.04\text{A}$). It was thought that the orientation of the aluminum might be determined by that of the chromium. To test this the orientation was determined for aluminum evaporated directly on glass, and was found to be the same as for aluminum on chromium. Apparently the relation between the distances is an accidental one.

The orientation of aluminum on various substrates has been investigated by Dixit⁴ and Bruck.⁵ They found that the temperature of the base seemed to be the most important factor in determining which plane was oriented parallel to it. Beeching³ found that the direction of evaporation was also a factor influencing orientation.

The patterns from the thin layers of aluminum were not significantly different from those produced by the thick layers. The rings were possibly somewhat more diffuse in the case of the thinner layers, a fact which might indicate smaller crystals.

The pattern obtained from the aluminum films was unchanged after exposure to air and after washing with water. Beeching³ has reported that he investigated the effect of washing with methyl alcohol, which he was told had a hardening action on a film of aluminum on chromium. He found no change in the diffraction pattern produced by the film after this treatment.

Since aluminum at room temperature is

believed to be covered with a protective oxide film, the fact that no evidence of the presence of any oxide was found in any of the patterns, even after exposure to air, is of some interest. There have been many attempts to detect this oxide film and to study its properties by electron diffraction. However, there seems to be little agreement as to the structure or thickness of the film. Although Dixit,⁴ Preston and Bircumshaw,⁶ Belwe,⁷ Steinheil,⁸ Bound and Richards,⁹ and Yamaguchi¹⁰ have studied reflection specimens of aluminum, only Beeching and Yamaguchi have obtained patterns which could be attributed to a form of aluminum oxide. The nature of the evidence Beeching found led him to the conclusion that the oxide he detected was formed when the specimens were evaporated, probably because of an oxygen layer on the surface of the glass. Yamaguchi, using an aluminum block, has found two faint rings which he attributed to aluminum oxide. In general the evidence would seem to show that the oxide film on aluminum is very thin and probably is amorphous, since only thus can the general absence of oxide rings in the diffraction patterns be explained. This conclusion is, however, not in agreement with the work of Tronstad and Höverstad.¹¹ They investigated the thickness of the oxide film on aluminum by an optical method and found that the film was 100 to 150A thick.

In conclusion, the author wishes to express her appreciation to Professor C. C. Murdock and Professor R. C. Gibbs for the helpful suggestions and criticism received during the course of the work.

⁴ G. D. Preston and L. L. Bircumshaw, *Phil. Mag.* **20**, 706 (1935).

⁷ E. Belwe, *Zeits. f. Physik* **100**, 192 (1936).

⁸ A. Steinheil, *Ann. d. Physik* **19**, 465 (1934).

⁹ M. Bound and D. A. Richards, *Proc. Phys. Soc.* **51**, 256 (1939).

¹⁰ S. Yamaguchi, *Inst. Phys. Chem. Res., Tokyo, Sci. Papers No. 942*, pp. 463-470.

¹¹ L. Tronstad and T. Höverstad, *Trans. Faraday Soc.* **30**, 362 (1934).

⁴ K. R. Dixit, *Phil. Mag.* **16**, 1049 (1933).

⁵ L. Bruck, *Ann. d. Physik* **26**, 233 (1936).