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Diffuse Electron Diffraction Patterns

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Diffuse electron diffraction patterns taken by reflection have in the past been cited as proof that the polished surfaces of metals are amorphous or very finely crystalline. In the present work it is shown that such patterns can be obtained from a large-grained crystalline surface and hence cannot be due to an amorphous phase but rather to the physical contour of the surface.

I. INTRODUCTION

ELECTRON diffraction patterns of the polished surfaces of metals and of some other crystalline materials show only two diffuse rings. By analogy with x-ray diffraction patterns of liquids and glasses this was assumed to prove the existence of an amorphous phase on the polished surface, as first suggested by Beilby.[1] However, Kirchner[2] and Germer[3] raised objections to this and gave alternative explanations of the observed patterns in terms of the physical contour of the surface and the refraction of the beam by the inner potential of the metal. In support of this they stated that very thin films which gave crystalline patterns by transmission yielded diffuse patterns by reflection. Independent evidence[4] and particularly some recent work by Cochrane[5] seem to indicate that the polished layer on metals is indeed either amorphous or very finely crystalline, and in the latter work the polished film is isolated and then examined by transmission so that the objections mentioned above do not arise.

Because of these objections it is now generally admitted that diffuse patterns when obtained by reflection only are not proof of the presence of an amorphous layer.[6] The following experiments support this conclusion since they show that such diffuse patterns may be obtained from samples containing relatively large crystals.

II. EXPERIMENTAL

The apparatus used was a hot filament type employing movable slits, similar to that described by Germer.[7] The sample-to-photographic plate distance was 69 cm and the accelerating voltage 30 kv.

The sample used was an alloy of iron containing 18 percent chromium, 8 percent nickel, and 0.08 percent carbon, commonly known as 18–8 stainless steel. It was fine-grained and wholly in the austenitic or face-centered cubic

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[1] G. Beilby, *Aggregation and Flow of Solids* (Macmillan Company, New York, 1921).

[2] F. Kirchner, Erg. exakt. Naturwiss. 112 (1932).

[3] L. H. Germer, Phys. Rev. **43**, 724 (1933); **49**, 163 (1936).

[4] G. I. Finch, A. G. Quarrell and J. S. Roebuck, Proc. Roy. Soc. **A145**, 676 (1934).

[5] W. Cochrane, Proc. Roy. Soc. **A166**, 228 (1938).

[6] G. I. Finch and S. Fordham, Chem. and Ind. **56**, 632 (1937); G. I. Finch, J. Roy. Coll. Sci. **7**, 32 (1937).

[7] L. H. Germer, Rev. Sci. Inst. **6**, 138 (1935).

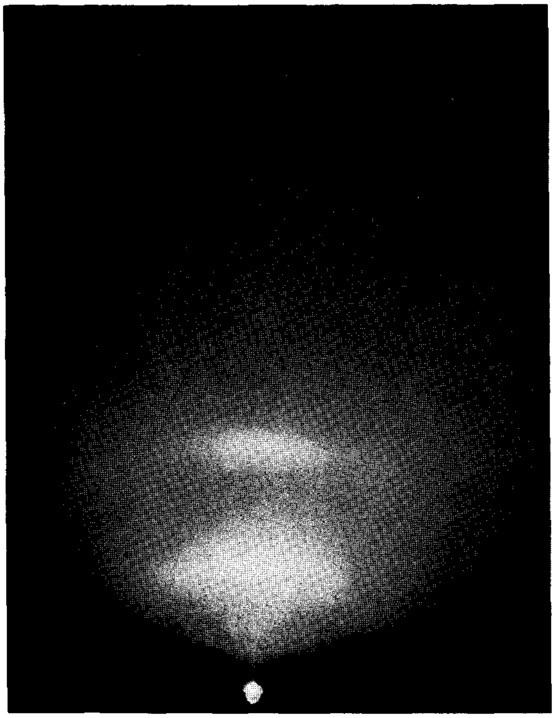

FIG. 1. Electron diffraction pattern of stainless steel polished under benzene.

phase before treatment. It was polished on various grades of emery paper down through 0000 and finally on chamois, all carried out under benzene. It was transferred while still wet to the diffraction camera which was immediately evacuated. This procedure was followed because, as shown by Dobinski,[8] samples polished in air may give a slightly different type of diffuse pattern which could be the result of exposure to the air, although lately, however, both the results and hypotheses of Dobinski have been questioned.[9, 10] The resulting diffraction pattern showed the two diffuse rings typical of such a polished surface (Fig. 1).

The specimen was then etched electrolytically in a 10 percent solution of oxalic acid until a depth, as estimated from previous quantitative electrolysis experiments, of 18,000A, was removed. At this depth all of the alpha- or body-centered cubic phase produced by polishing has been removed. The diffraction pattern of this surface is the face-centered cubic one typical of the alloy in question and is shown in Fig. 2.

The sample was then heated inductively to a temperature of 1000°C for half an hour in a glass vacuum system. The pressure before and after heating was 1.6×10^{-5} mm of Hg. Benzene was admitted to the system until the sample was covered by the liquid so that no air could reach it and it was again transferred wet to the diffraction camera. The resulting pattern (Fig. 3) was entirely similar to that of the polished sample although there was no question of such a layer being present in this case. Pronounced grain growth had taken place and the grain boundaries were clearly revealed. A photomicrograph of the surface is shown in Fig. 4.

The experiment was repeated but this time air instead of benzene was admitted to the sample after the vacuum annealing before it was placed in the camera. The pattern obtained was the same as that of Fig. 3 and is therefore not reproduced.

III. Discussion

This then is another example of a crystalline surface, in this case composed of relatively large crystals, which gives a diffuse pattern entirely similar to that from a polished surface of

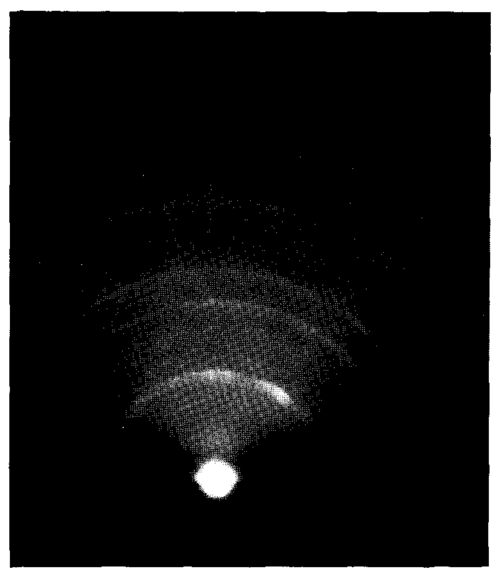

FIG. 2. Electron diffraction pattern of the same sample after being etched.

[8] S. Dobinski, Phil. Mag. **23**, 397 (1937).
[9] Campbell and Thomas, Nature **142**, 253 (1938).
[10] E. Plessing, Physik. Zeits. **39**, 618 (1938).

the same material. Finch and Ikin[11] have reported that prolonged heating in a vacuum broke down the crystalline structure of a particular platinum surface to an amorphous condition. It seems to the author that this deduction from their experiment is entirely untenable and that the explanation for the experiments reported here must be looked for elsewhere. It also seems unlikely that the pattern of Fig. 3 could be due to an adsorbed layer of benzene, since the air-exposed surface where no benzene was present gave precisely the same pattern. Furthermore such gas films would not be expected to give an appreciable pattern at 30,000 volts even on as flat a surface as this one is.

The physical contour of the surface is probably responsible for the observed pattern. In this experiment considerable evaporation of the surface took place before the pattern of Fig. 3 was obtained, and the sharp ridges considered so essential for a good diffraction pattern had disappeared. The surface remained somewhat wavy as seen in Fig. 4, but on the whole was flat enough so that the beam would enter and leave the metal through essentially the same surface although making different angles with this surface at different points on the sample. In this case refraction of the beam is appreciable and because of this varying angle it could distort and blur a crystalline pattern such as Fig. 2 considerably. This has already been suggested[2, 3] and can easily be the case here.

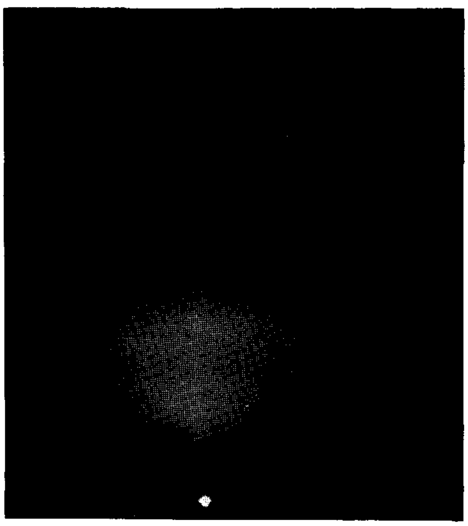

FIG. 3. Electron diffraction of the same after being vacuum annealed at 1000°C.

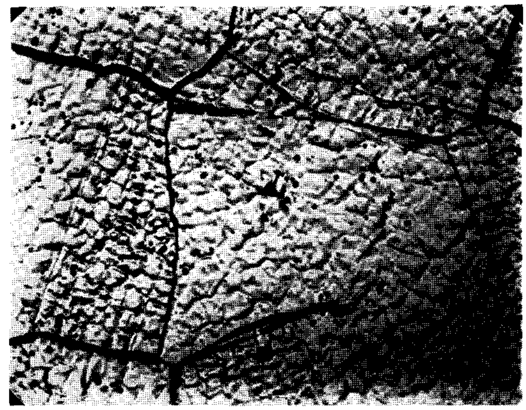

FIG. 4. Photomicrograph of vacuum-annealed surface of stainless steel, unetched, magnified 100×.

In any event this experiment shows that diffuse patterns, similar to those from the polished surface, can be obtained by reflection from large-grained crystalline materials, and hence such patterns when procured by reflection methods are not proof of the existence of an amorphous phase.

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[11] G. I. Finch and A. W. Ikin, Proc. Roy. Soc. **A145**, 559 (1934).