the Camp Ridge Quartzite indicate emergence and the development of topographic relief, and they may represent cessation of marine sedimentation and emergence of the whole area.

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Received April 10, 1972.

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The Direct Viewing and Brief-time Recording of Crystallographic Events

THE exposure times involved in obtaining X-ray diffraction patterns are an obstacle to the study of dynamic processes. Several workers have tried to devise visual presentations of the diffraction patterns from transient crystallographic events, and recently Chester and Koch1 reported some success using a vidicon tube which was sensitive to X-rays and which had a complex diode mosaic as photocathode. Other workers, including ourselves, have used a phosphor as an image converter, the visual image produced by the phosphor being amplified by a high-gain image intensifier. We have had some success with an image intensifier that has a television read-out, but the equipment is expensive, cumbersome, and has to be custom-built. Here we report that acceptable results can be obtained using relatively cheap standard night surveillance equipment currently being produced in the USA and available in the UK. These units are designed to be carried in the hand or even mounted on a rifle and are extremely compact and portable.

The 'Owl Eye' intensifier, for example, was developed by the Aerojet Delft Corporation of California for police work2. It gives a 125 mm diameter visual read-out which may readily be viewed by eye from a working distance of several feet and can be photographed with a standard camera. The result illustrated in Fig. 1 was obtained with this unit. X-rays from an 'Elliott GX6' generator operating at 40 kV and 50 mA were emitted through a 1.5 mm diameter exit port to give a slightly divergent beam. The specimen consisted of a thin copper grid used as the specimen support in electron diffraction work. This was positioned directly over the exit port, the main beam being trapped by a lead stop set in a brass saddle and the diffraction pattern being transmitted via lateral cutaways.

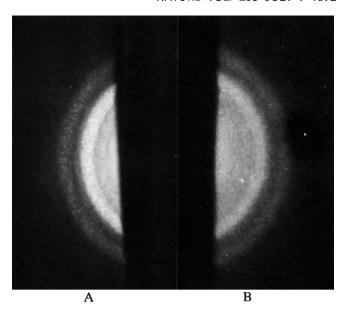


Fig. 1 Copper K interference pattern obtained from a polycrystalline copper specimen. A was recorded directly on fast X-ray film and required an exposure of 30 s. B was obtained by photographing a visual image on the image intensifier and required an exposure of 1/30 s.

To use the 'Owl Eye' image intensifier the X-ray diffraction pattern was first converted into a low light level visual image. This was done with a transmission fluorescent screen obtained from the trade and consisting of a silver activated zinc/cadmium sulphide phosphor (grade YSF) of particle size 25-30 μm. This is deposited with a suitable binder on to a thin melinex film to a density of about 35 mg per cm². The 'Owl Eye' system was then focused on this screen, viewing for reasons of radiation safety in a mirror set at 45°. Preliminary focusing was done with the X-ray set switched off and the room dimmed.

With the laboratory blacked out a bright image of the copper K_{α} reflexions from the (111) and (200) crystal planes of the copper specimen was readily obtained, the copper K_{β} reflexion from the (111) plane being just discernible. Using HP4 film developed in Microphen developer, and a 35 mm camera set at f2.0, a well balanced photographic negative was obtained in 1/30 s. A print of such an exposure is shown in Fig. 1 compared with a similar print taken from a direct X-ray exposure made on fast dental film in 30 s. Loss of detail is obvious, but this detracts only slightly from the value of the result as an effective brief time record.

With exposures of 1/30 s, the frame-by-frame recording by cine-camera of a sequence of short-lived crystallographic events becomes possible.

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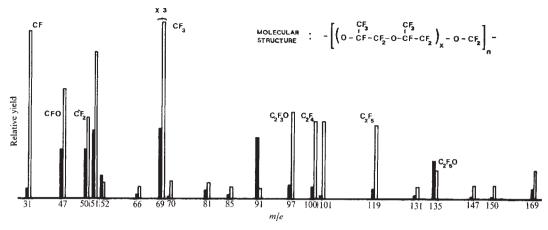
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Perfluoropolyether—a Vacuum Pump Fluid resistant to Electron Induced Polymerization

Growth of polymer films on the components of electron beam instruments is a problem experienced widely because of the extensive use of oil pumps on such apparatus. Recently, new thermally stable fluids have become available which are based

Fig. 1 Mass spectra of: black columns, atmosphere above the vapour stream pump at a chamber pressure of 2.5×10^{-6} torr; white columns, 'Fomblin' components admitted from source at 190° C and with chamber pressure of 2×10^{-6} torr.



on oxygen linkage of fluorocarbon groups; we have found that they can be used in vacuum pumps without the attendant polymerization problems. An earlier study¹ has shown that a high molecular weight (~6,500 a.m.u.) perfluoropolyether fluid has lubricating properties suitable for use in rotary pumps. The work indicated that a similar fluid of greater volatility (molecular weight ~3,000 a.m.u.) should be suitable for use in vapour stream pumps and here we give conclusive evidence of its value for this purpose. The fluids, known as 'Fomblin', are prepared for us by Montecatini Edison S.p.A. (see ref. 2).

The fluid to be tested was used in a vapour pump, Edwards EO4 5 inch diameter aperture, connected to a stainless steel chamber of 28 l. capacity. A rotary pump with a nominal speed of 35 l./min and charged with 'Fomblin' backed the vapour pump. The chevron baffle mounted in the vapour pump inlet was uncooled to ensure a high backstreaming level. The pressure measured by an ion gauge was 10^{-6} torr and the fluid was in use for a total of 1,500 h. We fitted an Edwards mass spectrometer (3 inch radius 60° magnetic sector field) with its unprotected (nude) ion source in the chamber, so that fragmentation spectra could be recorded for the vacuum atmosphere and vapour from the parent fluid injected close to the ion source.

Fig. 1 shows comparative spectra from a sample of unused 'Fomblin' evaporated from a side oven at 190° C and the backstreaming vapour from the vapour pump after 600 h use. Possible compositions of the molecular fragments are also given in Fig. 1. Mass spectra were generally measured up to an m/e ratio of 260, but the yield of fragments above 170 was small and we have not included them. Although the relative abundancies differ, the similarity between the molecular fragments in the two spectra suggests to us that they both arise chiefly by electron impact within the ion source of the mass spectrometer.

Further extensive tests were made by bombarding metal and glass slides with electrons for 1 h, first, in the presence only of backstreaming vapour from the pump, and second, with the fluid spread on the target. The electrons had an energy of 1 keV and a current of 0.5 mA was recorded with a target area of 25 cm². Occasionally, we could detect signs of polymer formation on the target but this was not unexpected because the gas analysis showed traces of hydrocarbons in the system from previous use of the pump with conventional fluids. But, as these impurities disappeared, so did the formation of polymers. The mass spectrometer study showed that electron impact will fragment the perfluoropolyether molecule, so we conclude that neither the parent molecule nor its fragments can be polymerized by electron bombardment either in the liquid or the adsorbed state.

We note that if a mass spectrometer with a nude source, as used in these experiments, had been operated at room temperature in the saturated vapour of any of the hydrocarbon or silicone pump fluids, then serious contamination and failure of the ion source would have ensued within a few hours.

The nude ion source of the instrument was operated in the saturated vapour of the fluid for 50 h and for a further 100 h in backstreaming components from the pump without deterioration in its performance.

Because the fluid can be used in rotary mechanical and vapour stream pumps it could prove valuable for pumping systems on electron microscopes and ion beam accelerators where secondary electrons abound. It will be necessary to ensure that molecular fragments of the fluid are not reactive with specimens or vacuum materials.

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Received February 3; revised March 24, 1972.

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Electrochemical Photolysis of Water at a Semiconductor Electrode

ALTHOUGH the possibility of water photolysis has been investigated by many workers, a useful method has only now been developed. Because water is transparent to visible light it cannot be decomposed directly, but only by radiation with wavelengths shorter than 190 nm (ref. 1).

For electrochemical decomposition of water, a potential difference of more than 1.23 V is necessary between one electrode, at which the anodic processes occur, and the other, where cathodic reactions take place. This potential difference is equivalent to the energy of radiation with a wavelength of approximately 1,000 nm. Therefore, if the energy of light is used effectively in an electrochemical system, it should be possible to decompose water with visible light. Here we describe a novel type of photo-electrochemical cell which decomposes water in this way.

Electrolysis of water can occur even without applying electric power if one of the following three conditions is fulfilled. First, oxygen evolution occurs at a potential more negative than that at which hydrogen evolution occurs in normal conditions; second, hydrogen evolution occurs at a potential more positive than that at which oxygen evolution occurs in normal conditions; third, the potential for oxygen evolution is made more negative and that for hydrogen evolution is made more positive, until the former is more negative than the latter.

Current-voltage curves of a semiconducting n-type TiO₂