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Film Deposits and Whisker Growth in the Electron Microscope

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ABSTRACT

Whisker growth has been observed on a soda-lime-silica glass during observation in an electron microscope under a high intensity beam. A similar growth was also noted on an electron microscope copper support grid which had been dipped in diffusion pump oil. Both the whiskers on the glass and the grid had identical electron diffraction patterns. It may be concluded that the whiskers are a decomposition product of the diffusion pump oil.

INTRODUCTION

That films deposit on certain materials when these materials are subjected to electron and ion bombardment in vacuo was well enough established by 1934 that R. L. Stewart devoted a paper to the phenomenon[1]. He concluded that the films were carbon compounds, that they resulted from the polymerization of organic vapors, and that they could be an insidious and prevalent source of experimental errors. Later, J. H. L. Watson reported on the deposit of such films with specific reference to the errors introduced in the measurement of particle size with the electron microscope[2]. Watson suggested that nonconducting materials may be relatively free of film deposits. The following is a report on film deposits encountered while investigating the structure of glass with the electron microscope. The deposits are of interest not only because they are a potential source of trouble in electron microscope studies of glasses but also because they can, under certain conditions, manifest themselves as whiskers. This whisker growth phenomena occurs under almost ideal conditions for observation.

ENVIRONMENTAL CONDITIONS FOR WHISKER GROWTH

The whisker growth phenomenon was encountered on both a Siemans Elmiskop-I and an Hitachi HU - 11 electron microscope. Of several glasses examined, whisker growth was noted only on the soda-lime-silica glass previously described by Levingood and Vong in connection with its interesting spiral defects[3]. Whisker growth was also noted to occur, in the absence of any glass, on a grid that had been dipped in diffusion pump oil.

The electron beam current was critical in encouraging whisker growth. On the Siemans microscope, with the beam sharply focused, a current of two to six microamperes was most favorable for whisker growth. For the Hitachi microscope the beam current ranged from 50 to 70 microamperes with the beam moderately sharply focused. (This discrepancy in beam current is not as great as it seems if the over-all performance of the microscopes is considered.) For both microscopes 100 KV electrons were used and the largest condenser aperture was in place.

OBSERVATIONS

Pertinent observations were as follows:

(a) Whisker growth was most pronounced near protuberances. Spikes of glass (see Fig. 1) are not necessary, but they do intensify and dramatize the effect.

(b) The whisker growth was influenced by the electron beam current and the beam focus. Extension of the whiskers could be halted by either lowering or raising the beam current.

(c) The maximum growth rate observed was about 50 Å/sec. (on the HU - 11).

(d) The whiskers, at their tips during growth, were as small as 20 Å across. This is a limiting and possibly a typical dimension. The conditions of observation did not always permit this low limit of resolution.)

(e) The whiskers were more resistant to high beam currents than the glass itself. Spikes of the glass softened at

beam currents (on the Siemens) of about 10 microamperes while matured whiskers were stable under beam currents of 25 microamperes.

(f) An example of whisker growth is shown in Fig. 1, and a more detailed sequence of a single whisker is shown in Fig. 2. As shown, the growth normally occurs in stages. The whiskers grow out up to a micron in length, tapering to a point. Extension then ceases while the whiskers thicken and the tips round off. Then new growth is nucleated on the old whisker and the cycle is repeated. As many as four such cycles of growth have been observed.

(g) Whiskers avoid each other by ceasing to extend or by curving (Fig. 3).

(h) Selected area diffraction patterns were made of the whiskers and of the glass. The whiskers yielded two diffuse rings typical of noncrystalline materials. These rings, of approximately equal intensity, indicated distances of 1.17 and 2.03 Å. The glass yielded a three ring pattern (strong, weak, very weak) indicative of distances of 2.90, 1.16, and 1.55 Å, respectively. A reasonable estimate of the accuracy of these measurements may be ± 5%.

(i) Vacuum pump oil, when diffracted, yielded a diffraction pattern identical to that of the whiskers.

DISCUSSION

Composition of the whiskers: From the diffraction data it can be concluded that the whiskers are a decomposition product of the diffusion pump oil.

Mechanism of growth: The whisker growth sequence of Fig. 2 shows that growth is not caused by extension at the base, since the fork of a whisker does not move out from the base with growth. Two other possibilities are that the whiskers grow by vapor condensation or by surface diffusion. Growth of whiskers by condensation of vapors is frequently associated with dislocation mechanisms in crystalline materials. As the whiskers are as small as 20 Å across at the tip it is difficult to clearly distinguish between the glassy and crystalline state. However, the gradual curvature of some whiskers, their frequent random forking at the tips, the general lack of angularity, and the diffuse nature of the diffraction pattern all suggest a noncrystalline growth mechanism.

A stalactitic type of growth would appear to be compatible with the observed whisker behavior. When the film is first deposited on the specimen it should retain some of the fluid properties of the diffusion pump oil. The driving force for whisker growth, which is presumably the mutual repulsion of positive ions of the film and their attraction to the electron beam, would force the film to the edges of the specimen. At the edge, local charge concentrations could nucleate whiskers. Growth of whiskers would then be dependent on the volatilization of one of the constituents of the viscous film or the polymerization of the constituents so as to leave a strong skeletal structure.

The growth of whiskers in stages indicates that a certain charge concentration is necessary at the tip in order to string out molecules. If this charge concentration is not maintained, the surface tension of the film becomes dominant, the tip rounds off, and extension ceases.

With inevitable fluctuations in the beam current of the microscope such behavior would explain growth in stages.

General remarks: The beam current at which spikes of the glass softened (about 10 microamperes) and the softening temperature of the glass in a furnace (roughly 800°C) place the temperature of the specimen in the 200 to 600°C range during whisker growth. Whisker growth was not noted on thin films of glass (obtained by blowing a bubble) but this may merely indicate that thin films are at a different temperature than the thicker chips when exposed to the same beam currents. That whisker growth was noted on only one type of glass chip may indicate an affinity of the film deposits for this glass. Such an affinity of the film deposits for carbon black was noted in footnote 2.

All glasses examined (fused silica, pyrex, sodium disilicate glass, ruby glass are a few) yielded a diffusion ring indicative of a distance of about 1.2 Å. While this is undoubtedly a result of a common dimension in the structure of these glasses, it is also a distance characteristic of the decomposition product of the diffusion pump oil.

CONCLUSIONS

(1) The material of the whiskers was identified as a decomposition product of the diffusion pump oil.

(2) A stalactitic mechanism of whisker growth is consistent with the observed behavior.

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FOOTNOTES

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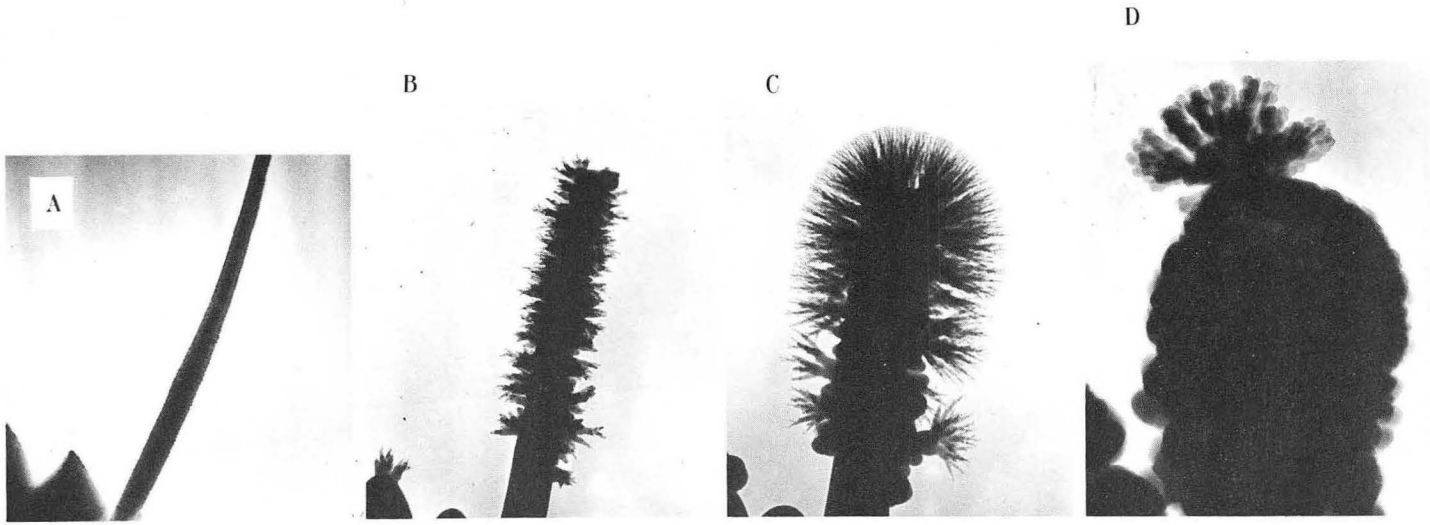

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Fig. 1. Spike of glass (A) with subsequent whisker growth (B-D). (A) 10,000 X, (B and C) 12,000 X, (D) 50,000 X.

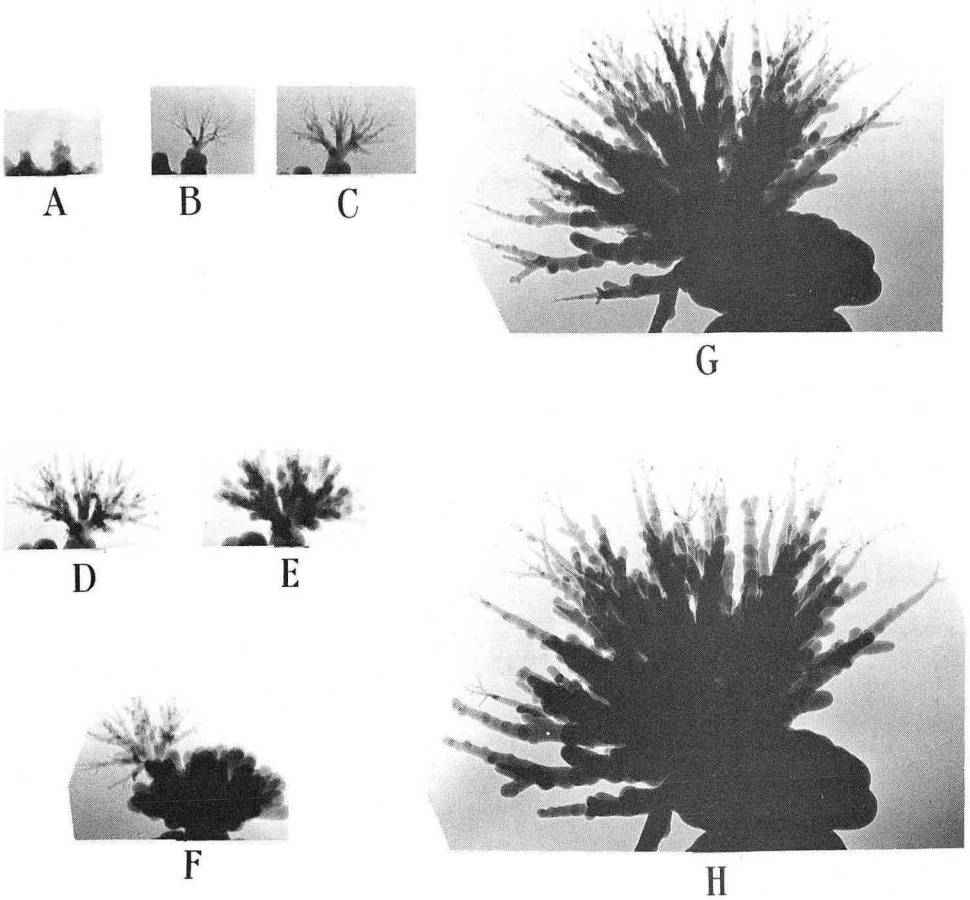

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Fig. 2. Growth sequence of a single whisker. The growth rate was relatively slow, about two hours being required for the sequence. (A-F) 20,000 X, (G and H) 40,000 X.

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Fig. 3. Detail showing how whiskers curve to avoid contact. 120,000 X.

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