SovietRxiv

Soviet-era science, translated into English

I. G. STOYANOVA, T. P. MOROZOVA

1963

SovietRxiv

View the original and related papers at https://sovietrxiv.org/items/ru-196301.05783

Source: Math-Net.Ru and CyberLeninka. Machine translation. Verify with the original.

SovietRxiv

Abstract

Full Text

I. G. STOYANOVA, T. P. MOROZOVA

STUDY OF DAMAGE ARISING IN ELECTRON-MICROSCOPIC OBJECTS UNDER THE ACTION OF ELECTRONS

(Presented by Academician A. A. Lebedev, 17 VIII 1962)

To preserve objects studied in an electron microscope in a viable state, it is necessary to apply a number of protective measures, including the introduction into the microchamber containing the object of a gas, with variation of its composition and pressure ([1,2]). The aim of the present work is to estimate the dependence of structural damage arising in electron-microscopic objects under the action of electrons on the conditions in which the irradiated specimen is placed.

It is known that in electron instruments (electron microscopes, electron diffraction cameras) operating at medium accelerating voltages, the structure of high-molecular compounds and biological objects is in most cases disrupted under the action of electrons ([3]), and these changes sometimes occur so rapidly that it is not possible to observe and record the electron diffraction pattern of the undamaged object. This circumstance once caused discrepancies between electron-diffraction data and X-ray structural-analysis data for some high-molecular compounds ([4]).

The investigation of the influence of various physical conditions, in which an electron-microscopic object placed in a gas microchamber is found, on the rate of occurrence in it of radiation damage under the action of electrons was carried out on single crystals of low-pressure polyethylene. Such single crystals were obtained from highly dilute solutions of polyethylene in benzene at a temperature of 70°. The specimen was prepared by depositing polyethylene single crystals, suspended in a drop of benzene, onto one of the diaphragms of the microchamber. A narrow electron beam (diameter 15 μ) was used, irradiating a small region of the object. As a result of electron diffraction from the single crystals, an electron diffraction pattern with bright interference maxima is formed on the microscope screen. As a result of the action of electrons on the object, the crystalline structure is disturbed, which appears in the electron diffraction pattern as a decrease in the intensity of the interference maxima, followed by their complete blurring and the appearance of three diffuse halos. The diffraction pattern was photographed with constant exposure at definite time intervals, the first exposure being made immediately after the electron beam was switched on. The intervals between exposures were chosen so that it was

SovietRxiv

Fig. 1

Figure 1: Fig. 1

Fig. 2

Figure 2: Fig. 2

possible to make several exposures from the moment irradiation began until the interference maxima disappeared completely. The electron diffraction patterns obtained were photometered along the direction of the reflections corresponding to the 110 planes. The photographs were made with different compositions and pressures of the gas filling the microchamber, and with different current densities and accelerating voltages.

For comparison of the damage arising in the object under different irradiation conditions, a coefficient was introduced characterizing the disturbance

of the object' s crystalline structure,

$$R = 1 - I(t)/I(t_0),$$

where $I(t)/I(t_0)$ characterizes the fraction of the intensity of an interference maximum at the irradiation time t relative to the intensity of the same interference maximum at the initial irradiation time t_0. $I(t)$ and $I(t_0)$ are determined from microphotograms of electron-diffraction patterns and are expressed in relative units,

$$I(t) = (D_{\text{m}} - D_{\text{f}})/D_{\text{m}},$$

where D_{m} is the blackening density at the maximum of the interference reflection, and D_{f} is the blackening density at the minimum following the interference maximum.

Fig. 1. Dependence of R on irradiation time for an electron-microscopic object in helium. $V = 50$ kV.
1 and 2–$j = 10^{-5}$ A/cm^2; 3 and 4–$j = 3 \cdot 10^{-5}$ A/cm^2; 5 and 6–$j = 10^{-4}$ A/cm^2. Values of p: a–$3 \cdot 10^{-1}$ mm; b–50 mm; v–$2 \cdot 10^{-4}$ mm; g–50 mm; d–$3 \cdot 10^{-1}$ mm; e–$2 \cdot 10^{-4}$ mm; zh–50 mm; z–$3 \cdot 10^{-1}$ mm; i–$2 \cdot 10^{-4}$ mm.

In Figs. 1, 2, and 3 are presented the results obtained in studying the influence of the gas, its composition, and pressure on the magnitude of the damage R arising under the action of electrons in an electron-microscopic object in a microchamber with gas (t is the irradiation time of the object).

Fig. 2. Dependence of R on irradiation time for an electron-microscopic object in air. $V = 50$ kV.

SovietRxiv

Fig. 3

Figure 3: Fig. 3

1–$j = 10^{-5}$ A/cm^2; 2 and 3–$j = 3 \cdot 10^{-5}$ A/cm^2; 4 and 5–$j = 10^{-4}$ A/cm^2. Values of p: a–50 mm; b–$2 \cdot 10^{-4}$ mm; v–50 mm; g–$3 \cdot 10^{-1}$ mm; d–$2 \cdot 10^{-4}$ mm; e–$3 \cdot 10^{-1}$ mm; zh–50 mm; z–$2 \cdot 10^{-4}$ mm.

As follows from the figures, the magnitude of the structural damage arising when the specimen is irradiated in vacuum ($2 \cdot 10^{-4}$ mm Hg) is greater than when the specimen is irradiated in a gaseous medium, and in the general case depends on the pressure and kind of gas.

The difference between the magnitude of the radiation damage in an object in vacuum ($2 \cdot 10^{-4}$ mm) and in a gaseous medium is apparently due to the fact that electrostatic charges arising on the surface of a nonconducting object when electrons pass through it aggravate the damage caused in the object by the electrons of the transmitted beam.

The visible effect of charging of the specimen in vacuum confirms this assumption. The magnitude of the electrostatic charge that arises on the surface of a nonconducting specimen when it is irradiated with electrons in vacuum and in a gas is different. Under vacuum conditions the charges arising on the surface of the specimen can create a large electric-field strength, reaching 10^6 V/cm ([5]). When the specimen is irradiated in a gaseous medium, an electrostatic charge of such magnitude as in vacuum does not arise because of partial or complete compensation of it by ionized gas molecules, the number of which is proportional to the pressure and to the ionization cross section of the molecules of the given gas.

Fig. 3. Dependence of R on time for an electron-microscopic specimen located in helium and in air. $V = 100$ kV.
1–$j = 10^{-5}$ A/cm^2; 2 and 3–$j = 3 \cdot 10^{-5}$ A/cm^2; 4 and 5–$j = 10^{-4}$ A/cm^2. Values of p:
a–$p^{\text{air}} = 3 \cdot 10^{-1}$ mm; b–$p^{\text{air}} = 50$ mm; v–$p^{\text{He}} = 3 \cdot 10^{-1}$ mm; g–$p^{\text{He}} = 50$ mm; d–$p^{\text{air}} = 3 \cdot 10^{-1}$ mm; e–$p^{\text{air}} = 50$ mm; zh–$p^{\text{He}} = 3 \cdot 10^{-1}$ mm; z–$p^{\text{He}} = 50$ mm; i–$p = 2 \cdot 10^{-4}$ mm; k–$p^{\text{He}} = 50$ mm; l–$p = 2 \cdot 10^{-4}$ mm.

The agreement of the values of R obtained in vacuum and at a helium pressure in the microchamber of $3 \cdot 10^{-1}$ mm for all current densities (Fig. 1) shows that the number of ions produced is insufficient to compensate the electrostatic charge arising in the specimen. The considerable decrease in R when the pressure in the microchamber is increased to 50 mm, and its constancy upon a subsequent increase in pressure, indicate that the ions produced in helium at a pressure of 50 mm compensate the action of the electrostatic charge. The fact that increasing the air pressure from $3 \cdot 10^{-1}$ mm does not cause a change in the magnitude of the damage, as in the case of helium, makes it possible to assume that the number of ions produced in air at a pressure of $3 \cdot 10^{-1}$ mm is already sufficient

to compensate the electrostatic charge. To obtain the same number of ions in helium, the ionization cross section of whose molecules is approximately 20 times smaller than the ionization cross section of air molecules, it is necessary to raise the helium pressure in the microchamber correspondingly by a factor of 20, i.e., to 6 mm. Irradiation of the specimen under the same conditions in an inert-gas medium—argon, the ionization cross section of whose molecules is close in value to the ionization cross section of air molecules—showed that, other conditions being equal, less damage arises in the specimen if its irradiation is carried out in an inert-gas medium. This is due to the fact that, when specimens are irradiated in an air medium, radiation damage is aggravated by the active interaction of atmospheric oxygen with the specimen. The fact that for polyethylene single crystals 100 Å thick at 100 kV no dependence of the magnitude of the damage on the type of gas and pressure, beginning from $3 \cdot 10^{-1}$ mm, was observed may indicate that at 100 kV,

in a thin specimen the magnitude of the electrostatic charge is reduced so much that even the number of ions arising in helium at a pressure of $3 \cdot 10^{-1}$ mm is already sufficient to remove this charge from a specimen 100 Å thick.

Analysis of the dependence $R = f(t)$ shows that at $V = 100$ kV and a current density on the specimen of $3 \cdot 10^{-5}$ A/cm^2, during the first 30 sec of irradiation practically no damage to the specimen occurs. Then, as the irradiation time increases, the buildup of damage proceeds differently depending on whether the specimen under study is in vacuum or in a gas. The irradiation time up to the moment when appreciable damage to the specimen appears in vacuum is 30 sec at $V = 100$ kV and 10 sec at $V = 50$ kV. For a specimen located in an inert-gas medium, this time reaches 50–70 sec. For higher current densities at these accelerating voltages, the flat portion of the curve $R = f(t)$ is not observed. The discovered feature of the dependence $R = f(t)$ shows that, in order to limit damage arising in a specimen under the action of electrons, under certain conditions of investigation one may make use of the factor of short irradiation time for preparations that are unstable to the electron beam.

Thus, the magnitude of the radiation damage arising upon irradiation of organic specimens in vacuum ($2 \cdot 10^{-4}$ mm) is considerably greater than upon irradiation of specimens located in a gas atmosphere. The role of the gas apparently amounts to removal of the electrostatic charge arising in the specimen when it is irradiated by electrons. The different dependence of the magnitude of radiation damage to specimens on pressure for different gases is due to the difference in the ionization cross sections of the molecules of these gases. The magnitude of damage to specimens depends on the kind of gas. The smallest damage occurs when specimens are irradiated in an inert gas. The substantially greater damage arising when specimens are irradiated in air is due to the fact that the radiation damage produced in the specimen by the passing electrons is intensified by the active interaction of the gas with the specimen. There exist modes of operation of the electron microscope in which the factor of short irradiation time can be used to limit the damage arising in the specimen under the action of electrons.

SovietRxiv

In conclusion, the authors express their gratitude to Academician A. A. Lebedev for a number of valuable comments and to Yu. M. Kushnir for his interest in the work.

Received:26 IV 1962

CITED LITERATURE

1. I. G. Stoyanova, T. A. Nekrasova, DAN, **134**, 227 (1960).

2. I. G. Stoyanova, Izv. AN SSSR, ser. fiz., **25**, 701 (1961).

3. B. V. Deryagin, N. A. Krotova, *Adhesion*, Izd. AN SSSR, 1949; A. L. Zaides, I. G. Sinitskaya, DAN, **80**, 213 (1951); A. L. Zaides, I. G. Stoyanova, DAN, **107**, 711 (1956); I. G. Stoyanova, A. L. Zaides, Izv. AN SSSR, ser. fiz., **23**, 758 (1959).

4. V. L. Karpov, V. A. Kargin, ZhFKh, **23**, 1502 (1949); V. A. Kargin, V. L. Karpov et al., DAN, **101**, 707 (1955).

5. H. Mahl, W. Weitsch, *Naturwiss.*, **46**, 487 (1959).

Note: Figure translations are in progress. See original paper for figures.

Source: Math-Net.Ru and CyberLeninka. Machine translation. Verify with the original.