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FORMATION AND RECTIFYING PROPERTIES OF A BARRIER AT CONTACT

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Abstract: The rectifying properties of a diode made from a cupric selenide sample in contact with magnesium were examined under low frequency A. C. conditions. The observed time dependence of the current through the diode and its current-voltage characteristic were explained considering CuSe and Mg as components of a tarnishing reaction under the electric field. As a result of this reaction, a layer of highly resistive magnesium selenide appeared. The change of its thickness led to rectification. Derived equations describe the experimental facts with satisfactory agreement. The ionic conductivity of MgSe was found to be $2.3 \cdot 10^{-6}\,\Omega^{-1}\,cm^{-1}$ at room temperature.

1. Introduction

It has been known for a long time that contact between CuS or CuSe and Mg or Al has an asymmetrical current-voltage characteristic, or that it shows some kind of rectification. Most of the published investigations concerning this problem[1, 2, 3, 4] were either empirical in nature or aimed at the preparation of commercial rectifiers. However, due to the very small permissible reverse voltage and some unforming or creep processes, these rectifiers have not found a wider application. Nevertheless, the respective authors observed that in the course of low-frequency operation the contact between CuS or CuSe and some metals develops an interesting reversible current-voltage characteristic (see Ref.[5]). As far as we know, this hysteresis-like characteristic is still unexplained. Our interest in the problem was due primarily to the fact that the rectification cannot be ascribed to the bulk semiconductivity of the compounds mentioned before, because they exhibit metallic properties[6, 7].

In the present work we describe some new results of our investigations carried out on a CuSe-Mg contact where the effect is particularly marked.

Keeping in mind that a layer of MgSe forms between CuSe and Mg and that Cu_2Se and metallic copper appear simultaneously[3, 4], we concluded that a tarnishing reaction takes place and so we treated the current through the contact on the basis of some established laws governing tarnishing processes.

2. *Tarnishing reaction of CuSe with Mg*

Though this reaction is known in principle[3, 4], it was never fully explained because many facts about the compounds which take part in the reaction are found only recently. To understand the barrier formation better, let us consider the reaction in detail.

If we put together a CuSe sample and a piece of Mg wire and switch this diode to the source so that Mg is positive (reverse direction), the initial current will be strong (the resistivity of CuSe at room temperature is only about $10^{-4}\,\Omega\,cm$[7]). However, Mg atoms in the surface near CuSe soon start to react with it according to the following formula

$$CuSe + Mg = Cu^{++}Se^{--} + Mg^{++} + 2e = Mg^{++}Se^{--} + Cu^{++} + 2e, \qquad (1)$$

producing a single-molecular layer of MgSe. Extracted Cu^{++} ion may recombine with two electrons and precipitate in CuSe, but it may also react with another molecule of CuSe

$$Cu^{++}Se^{--} + Cu^{++} + 2e = Cu_2^{+}Se^{--}.$$

Due to the permanent stoichiometric deviation x of cuprous selenide[8], this reaction proceeds as

$$Cu^{+}_{2}\,Se^{--} = Cu^{+}_{2-x}\,Se^{--} + x\,h + x\,Cu,$$

giving a certain quantity both of non-stoichiometric and p-type cuprous selenide (with x holes) and of metallic copper.

Thus, in each case we have a layer of MgSe between Mg and (CuSe + Cu) or ($Cu_{2-x}Se$ + Cu). Like CuSe, cuprous selenide behaves as a metallic, or at least, semimetallic conductor (the resistivity of $Cu_{2-x}Se$ depends on x, but its order of magnitude is $10^{-3}\,\Omega\,cm$[9]). However, magnesium selenide has nearly perfect isolating properties (the energy gap of MgSe being 5.6 eV[10]), and a layer of it, even if of a very small thickness, drastically reduces the electronic current through the diode.

At the same time, the ionic conduction of MgSe makes possible further growth of the layer. Supposing that the charge is transported exclusively by

Mg^{++} ions, the reaction $Mg = Mg^{++} + 2e$ takes place at the contact between Mg and MgSe. At the other end of MgSe, we have either the reaction (1) or the following reaction:

$$Cu^{+}_{2-x}\,Se^{--} + x\,h + x\,Cu + 2\,e + Mg^{++} = Mg^{++}\,Se^{--} + 2\,Cu.$$

When the electronic conductivity of MgSe is very small, the electrons taking part in both reactions can be supplied through the outer circuit.

As can be seen these reactions result in an increasing thickness of the MgSe layer and in an increasing quantity of precipitated copper. The growth of the layer can continue as long as the precipitated copper breaks off direct contact between MgSe and CuSe or $Cu_{2-x}\,Se$. Therefore, a layer of MgSe cannot grow infinitely, but can only reach a certain thickness L_m. We suppose that all the described reactions will take place in the reverse di-

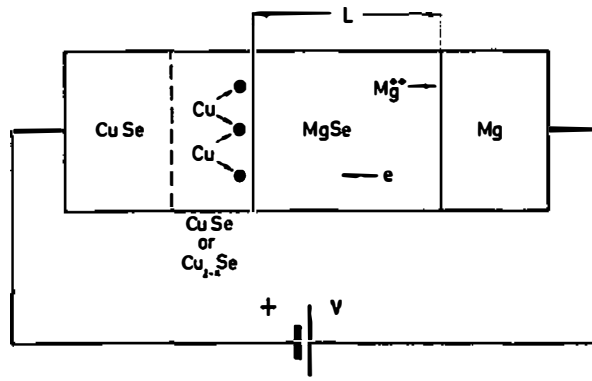

Fig. 1. Growth of the MgSe layer

rection if the current is reversed (forward direction). In this case the thickness of the layer decreases until the complete disappearance of MgSe and until direct contact between CuSe and Mg is established. For the sake of generality we must also permit that a spontaneous tarnishing reaction may be taking place even without an electric field.

Applying Ohm's general law to a layer of MgSe under the conditions shown in Fig. 1, one obtains the total current density j through the diode

$$j = -\sigma_i \frac{d}{dx}\left(\frac{\mu i}{2e}\right) - \sigma_i \frac{d\Phi}{dx} - \sigma_e \frac{d\Phi}{dx},$$

where σ_i and σ_e are average and coordinate independent ionic and electronic conductivities of MgSe, μ_i is the chemical potential of Mg^{++} ions, Φ the electrostatic potential and e the electronic charge. After a series of transformations[11] j reaches the form

$$j = \frac{\sigma}{L}(V - \frac{\sigma_i}{\sigma} V_s), \tag{2}$$

in which σ is the total conductivity of MgSe, L the layer thickness, V the external voltage and V_s the so-called stop voltage, at which the spontaneous tarnishing reaction is checked. At this voltage, the diffusion curent of Mg^{++} ions cancels the drift current of those ions. The ionic part of the total current causes the growth of the layer and density j_g of this part has the form

$$j_g = \frac{\sigma_i}{L}(V - V_s). \tag{3}$$

Finally, the growth rate is defined by the equation

$$\frac{\mathrm{d}L}{\mathrm{d}t} = -\frac{M}{2\rho F} j_g, \tag{4}$$

in which M is the molecular mass of MgSe, ρ its density, and F the Faraday constant. This equation represents the well known parabolic law of tarnishing reactions.

3. *Contact under A. C. voltage*

We tried to describe the behaviour of the CuSe—Mg diode under low frequency A. C. voltage by the Equs. (2), (3) and (4) introducing an A. C. voltage $V = V_m \sin \omega t$. Due to the high resistivity of MgSe and the expected

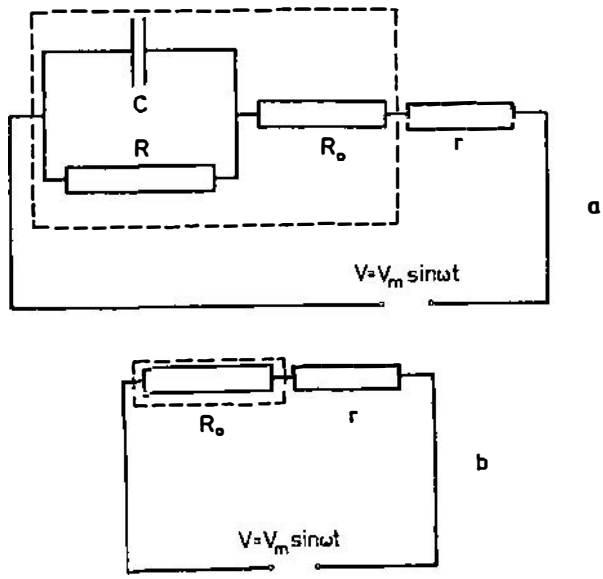

Fig. 2. Equivalent circuits of the CuSe-Mg diode

thin layer of it, the diode has certain capacitive and resistive properties, i. e. it may be represented by the equivalent circuit shown in Fig. 2a. Capacity C and resistance R are frequency independent. They change periodically since the thickness of the layer also varies periodically following the differential equation (4). R_0 represents the resistance of the diode if the layer thickness L is equal zero, while $r = 1\,\Omega$ is a standard resistor by which we obtain a voltage proportional to the current through the diode.

The experiments described below show that in certain cases the resistances R_0 and r play a role of minor importance. This is understandable considering very high resistivity of MgSe. Let us therefore consider the approximation $(R_0 + r) \ll R$. The current density derived from the equivalent circuit modified in this way is

$$j_e = \frac{\sigma}{L}\frac{V_m}{\cos\varphi}\sin(\omega t + \varphi) + \frac{\sigma_i}{L^3}\frac{\varepsilon M V_m^2}{2\rho F}\sin^2\omega t, \tag{5}$$

where φ is the phase angle given by the formula

$$\operatorname{tg}\varphi = R C \omega = \frac{\varepsilon\omega}{\sigma}, \tag{6}$$

in which ε is the dielectric constant of MgSe. Current density (5) is evidently due exclusively to external voltage. The total current density is obtained by adding to (5) the part which is due to the spontaneous tarnishing reaction. That part follows from (2) inserting $V = 0$. Thus, we can write

$$j = \frac{1}{L}\left[\frac{\sigma V_m}{\cos\varphi}\sin(\omega t + \varphi) - \sigma_i V_s\right] + \frac{\sigma_i}{L^3}\frac{\varepsilon M V_m^2}{2\rho F}\sin^2\omega t. \tag{7}$$

However, this is the sum of resistive and capacitive current densities. The growth of the layer is caused only by the ionic part of the resistive current. Consequently

$$j_g = \frac{\sigma_i}{L}(V_m \sin\omega t - V_s).$$

This equation, by help of (4) and integration from zero moment to some other moment t, gives the layer thickness as

$$L = \sqrt{L_m^2 + \frac{M\sigma_i}{\rho F}\left[V_s t - \frac{2V_m}{\omega}\sin^2\frac{\omega t}{2}\right]}. \tag{8}$$

Here L_m is an integration constant. Inserting the layer thickness (8) into (7), we obtain the final equation for the time dependence of total current density.

4. *Current through the contact*

Already a rough oscilloscopic observation of the current through the diode shows that it is a periodic and almost sinusoidal function of time (see Photo 1.). If we wish to describe this function by the equations (7) and (8), they must not contain time t explicitly and the influence of the second member in the equation (7) must be very small. The problem can be solved in the simplest way suposing firstly $V_s = 0$. This is a very plausible supposition

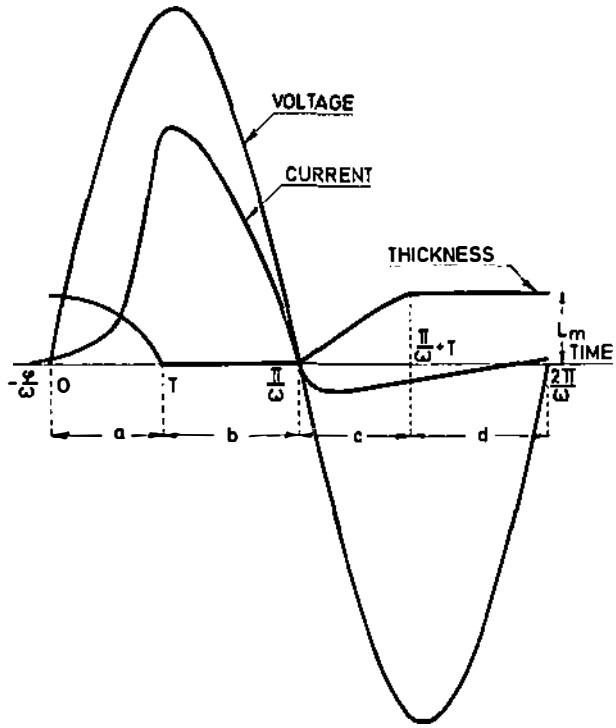

Fig. 3. Time dependence of the voltage, current, and layer thickness

because it only suggests the fact that no spontaneous tarnishing reaction takes place between CuSe and Mg, i. e. that layer of MgSe grows only if the current through the contact passes in the proper direction. Almost sinusoidal shape of the current enables us to consider the second member of (7) as equal to zero, at least as a rough approximation. Accepting these suppositions, we obtain from (7) and (8)

$$I = \frac{\sigma}{L}\frac{S V_m}{\cos\varphi}\sin(\omega t + \varphi), \tag{9}$$

$$L = \sqrt{L_m^2 - \frac{2 M \sigma_i V_m}{\rho F \omega}\sin^2\frac{\omega t}{2}}\,, \tag{10}$$

where I is the total current through the diode and S the cross-section of the MgSe layer. The constant of integration L_m has now the meaning of the maximal layer thickness. For »small« peak voltages V_m the current I has indeed a sinus shape and is shifted by φ against the voltage V.

The peak voltage has these »small« values as long as the equation (10) gives for L a real quantity. When the peak voltage reaches the value $V_m = V_{ms}$, the layer thickness L disappears at each moment $t = (2n + 1)\pi/\omega$. This limiting peak voltage is evidently defined by the equation

$$V_{ms} = \frac{L_m^2 \rho F \omega}{2M\sigma_i}. \tag{11}$$

Further, for $V_m > V_{ms}$ equation (10) gives for L imaginary values in definite time intervals.

Let us analyse this case in detail:

— at the moment $t = 0$ the layer thickness has its maximal value L_m. As time proceeds, the layer decomposes to disappear at the moment $T = T(V_m)$ (Fig. 3a). Thus, in the time interval $0 \leqslant t \leqslant T$ the layer thickness is given by (10) and the current through the diode by (9);

— in the second interval, $T \leqslant t \leqslant \pi/\omega$, equation (10) gives imaginary values for L. The physical meaning of all these values is $L = 0$ (Fig. 3b). There is no layer of MgSe, and the equivalent circuit of the diode in this case is shown in Fig. 2b. It follows that the current through the diode has the form

$$I = \frac{V_m \sin \omega t}{R_0 + r}. \tag{12}$$

Strictly speaking, if L tends to zero the approximation $(R_c + r) \ll R$ becomes formally less and less valid and in the same time the second member in (7) cannot be formally ignored. However, experiments show that (12) just follows (9). This means that MgSe layer exhibits a very high resistance, even if L is extremely small.

— a new interval of time begins at $t = \pi/\omega$. Equation (10) also gives imaginary values, but this is physically impossible because voltage polarity again ensures the conditions required for the growth of a new layer of MgSe (Fig. 3c). Integrating equation (4) from π/ω to some arbitrary moment t we obtain

$$L = \sqrt{\frac{2M\sigma_i V_m}{\rho F \omega}} \cos\frac{\omega t}{2},$$

$$I = \frac{\sigma S}{\cos \varphi} \sqrt{\frac{\rho F \omega V_m}{2 M \sigma i}} \frac{\sin (\omega t + \varphi)}{\cos \omega t/2}. \tag{13}$$

— the preceding time interval ends at the moment $t = \pi/\omega + T$, at which L becomes L_m. Mathematically, we denote L_m as an integration constant with a meaning of maximal layer thickness. In Chapter 2 we have also mentioned that the layer actually can grow only to a certain extent. This is also a plausible conclusion. The law for $\mathrm{d}L/\mathrm{d}t$ was derived from a general type of tarnishing reaction where the layer of a compound can grow infinitely. For example, the oxygen atmosphere in which a metal plate oxydises provides an infinitely rich source of oxygen ions, and the tarnishing reaction can persist. Between CuSe and Mg the reaction of another nature takes place. Here, CuSe acts as the donor of Se ions. It is not infinitely rich. The formation of the MgSe layer causes a simultaneous precipitation of Cu atoms. One can imagine that after a layer of a definite thickness is formed, the quantity of precipitated copper is so great that, in the form of a film, it breaks off the contact between CuSe and Mg, i. e. stops further reaction of CuSe with Mg. At this moment the layer of MgSe reaches its greatest thickness which we identify with the constant L_m. Now, we are able to see that assumption $V_s = 0$ play not an essential role. Even if $V_s \neq 0$, but small, the general shape of the function $I = I(t)$ will be preserved. Only the critical time T may be influenced a little.

Therefore, in the fourth time interval, $(\pi/\omega + T) \leqslant t \leqslant 2\pi/\omega$, we have $L = L_m$ (Fig. 3d) and the current through the diode is

$$I = \frac{\sigma S V_m}{\cos \varphi} \cdot \frac{\sin (\omega t + \varphi)}{L_m}. \tag{14}$$

Fig. 3 shows the current in these four intervals of time represented in arbitrary units and constructed according to equations (9), (12), (13) and (14). Within the interval where $L = 0$ the current is much greater than it is in the intervals where $L \neq 0$. This is in accordance with the approximation $(R_0 + r) \ll R$.

5. *Current — voltage characteristics*

For the peak voltages $V_m < V_{ms}$ the I—V characteristic of the CuSe-Mg diode is given in parametric form by the current (9) and the voltage on the diode which, due to the approximation $(R_0 + r) \ll R$, is identical with the voltage of the source, i. e. $V = V_m \sin \omega t$. Combining these two equations, we obtain

$$I = \frac{V \pm \sqrt{V^2_m - V^2}\,\mathrm{tg}\,\varphi}{\sqrt{L^2_m - \frac{M\sigma_i}{\rho F \omega}(V_m \mp \sqrt{V^2_m - V^2})}}. \tag{15}$$

This expression shows that the shape of the current-voltage characteristic changes markedly with applied peak voltage. A rather complicated analysis reveals that two basic shapes may be encountered. For very low peak vol-

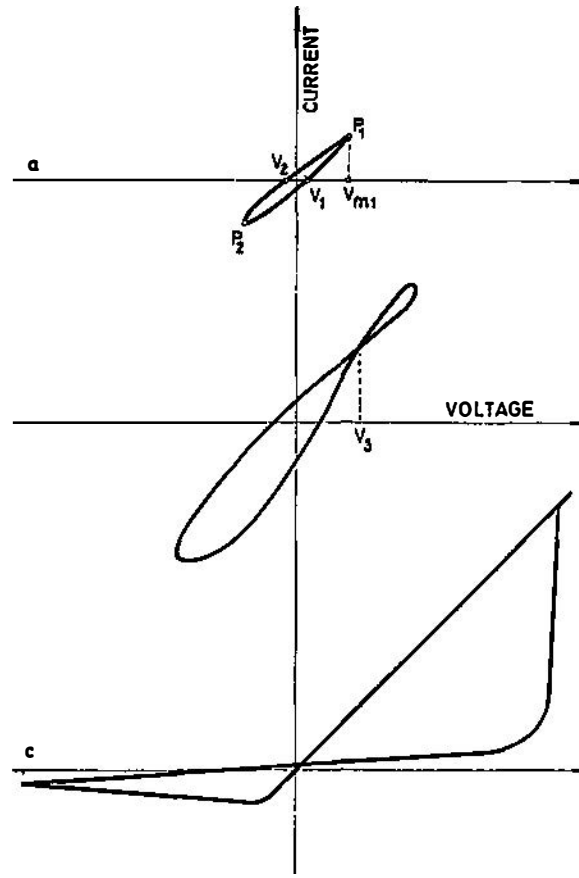

Fig. 4. Current-voltage characteristics at various peak voltages

tages the I—V characteristic is a narrow elliptical curve represented in arbitrary units in Fig. 4a. If the peak voltage increases and exceeds a certain critical value V'_{ms} the curve obtains a knot in the first quadrant (Fig. 4b). With increasing voltage the knot shifts to the origin and for $V_m = V_{ms}$ it is clossest to zero.

The most interesting feature of the I—V characteristic is the fact that for $V_m = V_{ms}$ it changes its shape suddenly. We know that equation (9) for $V_m > V_{ms}$ loses its meaning in certain intervals of time and therefore the

I—V characteristic (15) does not apply, either. We must construct a new characteristic with the voltage $V = V_m \sin \omega t$ and a current defined by Equs. (9), (12), (13) and (14). This characteristic in arbitrary units is represented in Fig. 4c.

6. *Introduction to the experiments*

The aim of the experimental part of this work was not only to show a general agreement between analytically derived curves $I = I(t)$ and $I = I(V)$ and curves found during the experiment, but also to evaluate certain parameters of a CuSe-Mg diode and MgSe compound. The first possibility is offered by oscilloscopic measurement of the voltages V_1 or $V_2 = -V_1$, which are placed symmetrically against the origin on an I—V characteristic for $V_m < V_{ms}$ (Fig. 4a). From (15) for $I = 0$ we obtain $V_1 = V_{m1} \sin \varphi$ which enables us, using the measured peak voltage V_{m1}, to determine the phase angle as

$$\varphi = \text{arc sin} \frac{V_1}{V_{m1}}. \tag{16}$$

One can measure the limiting peak voltage V_{ms} and the slope of a straight line through two points P_1 and P_2 on the same characteristic. After determining the corresponding currents for $V = \pm V_{m1}$ by (15) and using (11), we can obtain the slope $(\Delta I/\Delta V)_1 = \sigma S/\sqrt{L^2 \omega^2 - M \sigma_i V_m/\rho F \omega}$. This auxiliary equation gives

$$\sigma_i = \frac{M(2V_{ms} - V_m)}{S^2 \rho F \omega} \left(\frac{\Delta I}{\Delta V}\right)_1^2, \tag{17}$$

and

$$L_m = \frac{M \sqrt{2V_{ms}(2V_{ms} - V_m)}}{S \rho F \omega} \left(\frac{\Delta I}{\Delta V}\right)_1. \tag{18}$$

Both equations are derived assuming $\sigma_i \approx \sigma$, which is obvious for typically ionic MgSe.

Finally, the reciprocal slope of the I—V characteristic for $V_m > V_{ms}$ in the second quarter-period (Fig. 4c), where $L = 0$, is — according to (12) — $(\Delta V/\Delta I)_2 = R_0 + r$. Assuming $r = 1\,\Omega$ this gives directly

$$R_0 = \left(\frac{\Delta V}{\Delta I}\right)_2 - 1. \tag{19}$$

All these equations will find their application in the next Chapter.

7. *Experimental procedure and results*

The measurements were performed on the CuSe-Mg diode whose construction is shown in Fig. 5. It consists of a small cylindrical sample of CuSe which was joined with a piece of magnesium wire. The surface of CuSe was

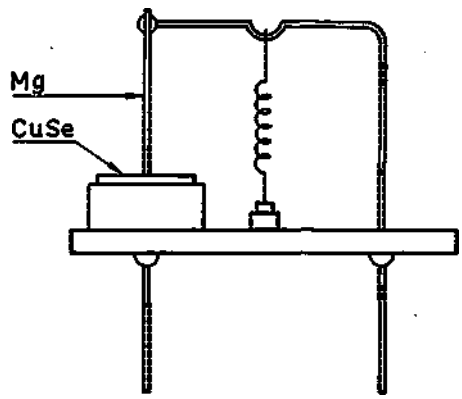

Fig. 5. Structure of the CuSe-Mg diode

finely ground and polished. The geometrical cross-section S of the contact was about 1 mm². The opposite, ohmic contact was made by means of galium on brass. Due to the chemical instability of MgSe in air the diode must be placed in a protecting environment. Best suited for this purpose is dehydratized toluene. The experimental facts about the preparation and examination of the electrical properties of CuSe was published elsewhere[7].

Most of the measurements on the CuSe-Mg diode were made by means of A. C. current of 50 cps, the most outstanding parameter of which, the limiting voltage V_{ms}, was previously determined. It was found to be $V_{ms} =$ $= 0.56$ V. Knowing this quantity, one can compare the experimental curves with those predicted by theory. Photo 1 shows the time dependence of the current through the diode at peak voltage $V_m = 0.11$ V. Since this value is below $V_{ms} = 0.56$ V, the current is almost sinusoidal, as expected. For a higher peak voltage, $V_m = 1.56$ V which is far above limiting voltage, the current has to be similar to that shown in Fig. 3. The oscilloscopic curve in Photo 2 shows that there really exists a certain likeness.

The agreement between the experimental and the derived curves is much more evident if one compares the I—V characteristics. For example, Photo 3 shows such a characteristic for $V_m = 0.15$ V. This value is below the critical voltage V'_m and therefore the photographed I—V characteristic corresponds to that shown in Fig. 4a. Further, for $V_m = 0.44$ V, which is greater than V'_m, one obtains the curve in Photo 4. Here, all conditions for a knot on the I—V characteristic were fulfilled and we have the case shown in Fig. 4b. Finally,

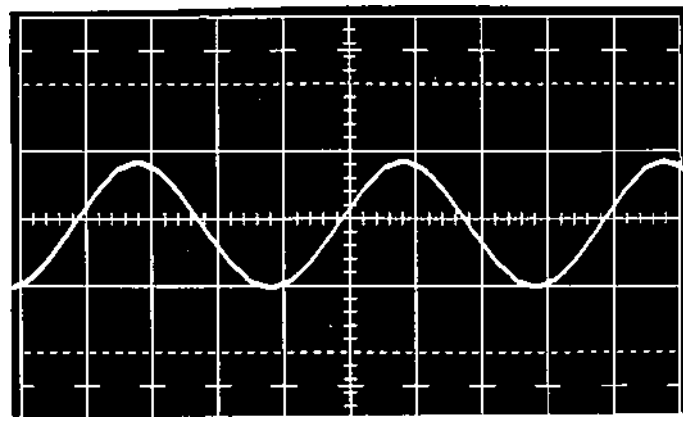

Photo 1. $I = I(t)$, $V_m = 0.11$ V, 50 cps, $y = 2$ mA/cm.

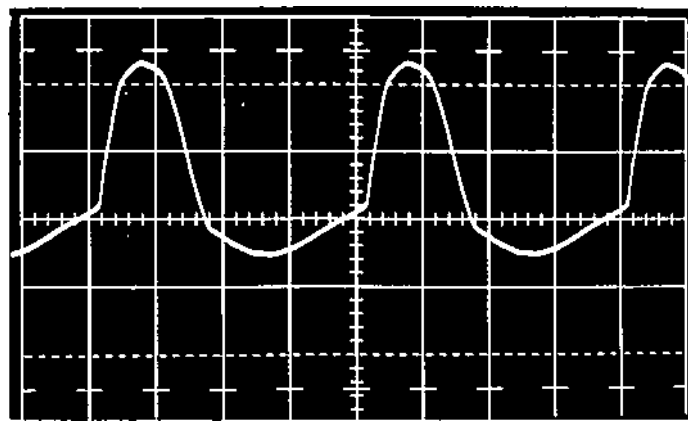

Photo 2. $I = I(t)$, $V_m = 1.56$ V, 50 cps, $y = 500$ mA/cm.

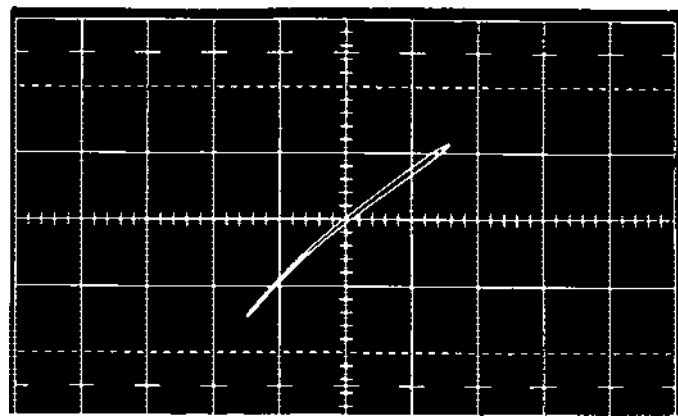

Photo 3. $I = I(V)$, $V_m = 0.15$ V, 50 cps, $x = 100$ mV/cm, $y = 2$ mA/cm.

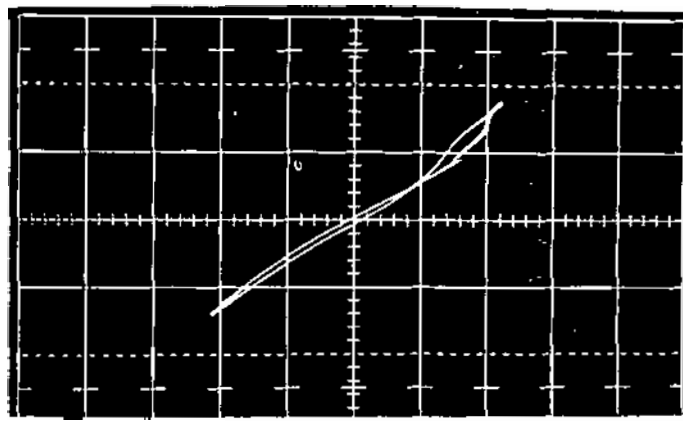

Photo 4. $I = I(V)$, $V_m = 0.44$ V, 50 cps, $x = 200$ mV/cm, $y = 4$ mA/cm.

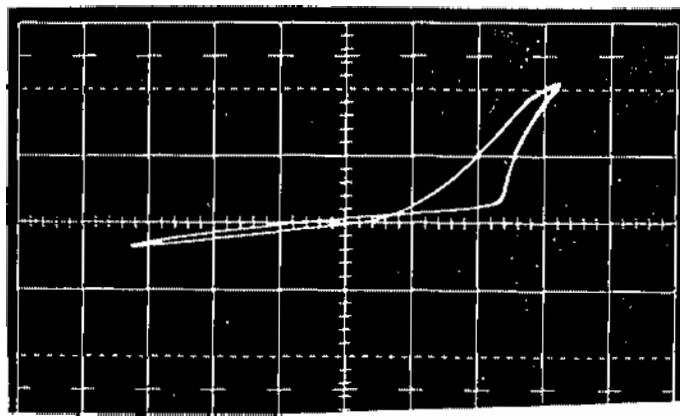

Photo 5. $I = I(V)$, $V_m = 1.65$ V, 50 cps, $x = 500$ mV/cm, $y = 100$ mA/cm.

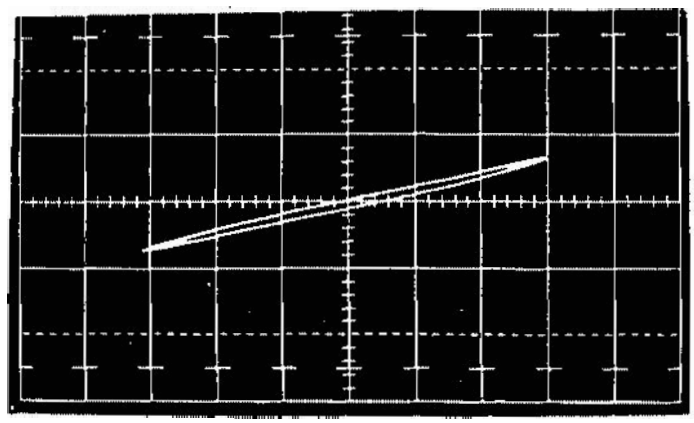

Photo 6. $I = I(V)$, $V_m = 1.50$ V, 15 000 cps, $x = 500$ mV/cm, $y = 500$ mA/cm.

for $V_m = 1.65$ V, the experimental curve is represented in Photo 5. If this curve is compared with that shown in Fig. 4c, one finds again an evident agreement.

Let us see now what the calculations showed. Our measurements at 50 cps, together with Equs. (16), (17), (18) and (19), gave the following results

$$\varphi \approx 0, \quad \sigma_i = 2.3 \cdot 10^{-6}\,\Omega^{-1}\,\mathrm{cm}^{-1}, \quad L_m = 1.4 \cdot 10^{-6}\ \mathrm{cm}, \quad R_0 = 0.3\ \Omega.$$

As far as we know, the value for σ_i mentioned above is the first published value for the ionic conductivity of magnesium selenide. The measurements of the phase angle were very inacurate. The phase angle seems to be very small and the nature of the sinus function near $\varphi = 0$ causes a great error in calculations using equation (16). Thus, the opportunity for a calculation of the dielectric constant of MgSe using (6) cannot be used. Thus we cannot establish accurately whether any unknown electrode processes take place in whole capacity of the diode. If they do exist, our model ignores them.

It remains still to describe the dependence of the effect upon frequency. Two parameters in the general equation (9) for the current through the diode are frequency dependent: L and $\cos\varphi$. If ω increases, L tends to L_m, and $\cos\varphi$ to zero. Thus, the current should increase with increasing ω, although the peak voltage V_m remains constant. Consequently, the I—V characteristic should change its shape with increasing frequency. First the change from Fig. 4c to Fig. 4a (due to L tends to L_m) should be observed. Then, the characteristic should broaden (due to increasing $\sin\varphi$) and its slope should increase infinitely due to decreasing $\cos\varphi$.

However, these events did not appear in the experiment. The reason for this is the fact that the approximation $(R_0 + r) \ll R$ becomes less and less valid as ω increases. Thus, we must take into account the complete equivalent circuit in Fig. 2a. The current (9) cannot increase infinitely: it increases only to the value given in equation (12). Therefore, the current-voltage characteristic becomes narrower rather than broader. Its slope tends to the value $1/(R_0 + r)$. This is nicely illustrated by Photo 6 in which the I—V characteristic for $V_m = 1.50$ V and 15 000 cps is represented. As can be seen, its shape is almost identical with that in Photo 3. However, both slopes are in complete disagreement, as it was expected. At 50 cps the diode has a mean resistance of about 60 Ω, which at 15 000 cps drops to about 4 Ω. The examinations at higher frequencies were not successful because the characteristic was gradualy deformed due to the influence of parasitic capacities in the measuring circuit.

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FORMIRANJE I ISPRAVLJAČKA SVOJSTVA BARIJERE NA KONTAKTU IZMEĐU CuSe I Mg

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Sadržaj

Rad opisuje rezultate istraživanja ispravljačkog efekta koji se javlja na kontaktu između bakar selenida i magnezija.

Prethodna i nedavno objavljena istraživanja električnih svojstava samog bakar selenida pokazala su da on ima svojstva karakteristična za metale. Ispravljački efekt mora se zato pripisati nekoj površinskoj barijeri.

Opažena vremenska zavisnost struje kroz kontakt i njegova strujno naponska karakteristika objašnjena je u radu na temelju zakonitosti kemijske reakcije između CuSe i Mg koja se zbiva pod utjecajem električnog polja. Kao rezultat te reakcije stvara se sloj magnezij selenida, MgSe, vrlo visokog električnog otpora. Promjene u debljini sloja uzrokuju efekt ispravljanja.

Izvedene relacije opisuju eksperimentalne krivulje sa zadovoljavajućom točnošću. One omogućuju izračunavanje ionske vodljivosti magnezij selenida. Dobivena vrijednost $2.3 \cdot 10^{-6} \Omega^{-1} \mathrm{cm}^{-1}$ predstavlja prvi objavljeni podatak o električnoj vodljivosti tog spoja.