Some electrical-contact effects that occur when a conical indenter is pressed into a flat anvil through a thin layer of dielectric material

S. M. Stishov and V. N. Kachinskii

Institute of Crystallography, USSR Academy of Sciences

(Submitted 31 October 1980; resubmitted 8 January 1981)

Pis'ma Zh. Eksp. Teor. Fiz. 33, No. 3, 136-140 (5 February 1981)

It is shown that the electrical effects observed during the compression of a thin dielectric layer in a system comprised of a conical indenter and a flat anvil can be described as the result of a reversible short circuit without invoking the hypothesis of a metal-dielectric junction.

PACS numbers: 77.90. + k, 85.20.Sn

An abrupt decrease in electrical resistance due to compression of thin dielectric layers between a conical indenter and a flat anvil, which were made from a synthetic diamond material—carbonado, was reported in Refs. 1–9. The return to the insulator state as the load is decreased occurs with a noticeable hysteresis, and the heating of a system near the return point gives rise to a spontaneous transition to the insulator state. Note that since the synthetic carbonado is a conductor the indenter and the anvil are used as electrical probes.

The described effects are interpreted by the authors as proof of the transition of the materials under investigation into the metallic state at high pressures. The group of materials, which have undergone metallization, is very large and includes hydrogen, water, diamond, boron nitride, aluminum oxide, alkali-metal fluorides, etc. Similar results were reported in Refs. 10–13 for silica, hydrogen, water, and magnesium oxide. Both groups of researchers indicate that the observed effects occur at a pressure of the order of 1 Mbar and higher.

Despite the fact that the described effects are very much like those expected to occur during first-order phase transitions, the interpretation of the obtained results as total metallization in this pressure range raises some doubts. Even if the pressure in the mentioned experiments reaches 5 Mbar, it is hard to assume that such poorly compressible substances as magnesium oxide, aluminum oxide, and silica become metals in this pressure range (see, for example, Ref. 14).

A question arises concerning an alternative explanation of the effects described in Refs. 1–13. For this reason, we have carried out experiments with an apparatus consisting of a conical indenter and flat anvil which were made from the VK-6 hard alloy. The yield stress due to compression of this alloy is equal to about 600 kgf/mm² (60 kbar); significantly higher pressures than those quoted, but apparently not exceeding 15 200 kbar, can be obtained by using the VK-6 material and special techniques. Therefore, the megabar range cannot be achieved by using this material.

The experiments were performed with indenters that had cone-vertex angles of 120° and 168°. The tips of the indenters were rounded off with a radius of ~ 0.2 –0.5

128

mm. The indenters were loaded by means of a screw-jack mechanism that is capable of producing forces up to 500 kgf. The test objects were thin layers of silica, aluminum oxide, and magnesium oxide. The electrical resistance between the indenter and the anvil was measured with a VK7-9 volt-ohmmeter. As it turned out, a suitable choice of insulator layer thickness made it possible to reproduce a load dependence of the electrical resistance that is almost identical to the corresponding dependences given in Refs. 1–9 (see Figs. 1 and 2).

We shall describe the experiments with magnesium oxide in more detail. The material was deposited on the anvil by burning metallic magnesium in the air. To obtain the desired effect, the optimum layer thickness had to be ~ 0.01 mm. At a layer

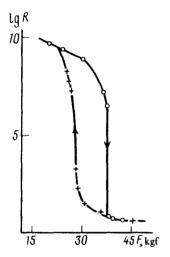


FIG. 1. Load dependence of electrical resistance for aluminum oxide. The indenter tip angle is 120°. The average pressure at the point of abrupt decrease in resistance is ~ 50 kbar.

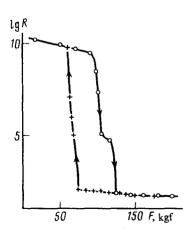


FIG. 2. Load dependence of electrical resistance for magnesium oxide. The indenter tip angle is 168°. The initial layer thickness is ~ 0.01 mm and the average layer thickness under indenter after several compression cycles is ~ 1 μ m. The average pressure at the point of abrupt decrease in resistance is ~ 30 kbar.

thickness much less than this value a short circuit occurred immediately after lowering the indenter to the surface. For a layer thickness $\gtrsim 0.03$ mm the desired effect did not occur even with the maximum loads. A typical dependence of the resistance R on the load F for magnesium oxide is shown in Fig. 2. Moreover, just as in the experiments of Refs. 1–9, heating the system at points close to the transition point results in a spontaneous transition to the nonconducting state. Light tapping produces a similar result. At points that are close to the transition to the conducting state light tapping often results in an abrupt decrease in the resistance.

In many cases the R(F) curves in the vicinity of the abrupt change in conductivity have a multistep form. Sometimes the abrupt change of resistance at the transition point is initiated by a change in the measuring current and voltage; this indicates that there is an electrical breakdown.

Measurements of the area of the indenter impression on the magnesium oxide layer and the force, which is produced by the press, give an average pressure of ~ 30 kbar at the point of abrupt decrease in resistance. Even if we assume that the pressure at the center is several times greater than the average pressure, we would have to be very optimistic to assume that it reaches 1 Mbar and higher. On the other hand, it is known that magnesium oxide exhibits no signs of metallization up to pressures of 1 Mbar. ¹⁶

The foregoing discussion applies to our experiments with aluminum oxide (see Fig. 1). The average pressure in this case was equal to ~ 50 kbar, whereas it has been shown experimentally ¹⁷ that aluminum oxide remains an insulator up to pressures of ~ 1.7 Mbar.

The observed effects can be explained in terms of a reversible shorting of the indenter and anvil through the thin insulator layer. A microscopic examination of the compressed material shows that the initially loose material was highly compressed and became transparent. The thickness of the layer of material under the central part of the indenter is $\sim 1~\mu m$, on the average; this is comparable to the size of some of the irregularities on the indenter and anvil surfaces.

A shorting mechanism can be the penetration of protrusions of the indenter and anvil through the intergranular spaces and cracks in the thin insulator layer due to nonuniform elastic deformation; in this case, the overall picture of mechanical shorting may be complicated by electrical breakdown or by quantum-mechanical tunneling effects, depending on the voltage applied to the sample.

The observation of electrical breakdown in many of our experiments makes it possible to estimate the thickness of the tested material at the point of breakdown. The voltage across the sample can reach 6 V during the measurement of resistance by the VK7-9 instrument. The electrical-breakdown voltage for materials such as A1₂O₃,MgO, and SiO₂ in thin layers is $\sim 5 \times 10^6$ V/cm. Consequently, the thickness of the layer of material at the breakdown point is ~ 100 Å for an average layer thickness of $\sim 1~\mu$ m. Since the surface roughnesses of the anvil and indenter are much larger than 100 Å, we see no reasons preventing a direct mechanical contact between the indenter and the anvil with further increase of the load.

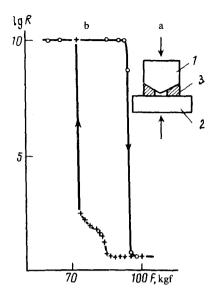


FIG. 3. (a) Mechanical model illustrating a reversible short circuit through a viscoelastic medium. 1, Metallic cone; 2, metallic anvil; 3, Kapron washer; (b) load dependence of electrical resistance for the model illustrated in Fig. 3a.

The observed hysteresis can be accounted for by the large losses due to friction and plastic deformation of the material. The return to nonconducting state due to heating and vibration or the reverse transition due to the influence of these factors is attributable to a number of reasons, such as the reduction of friction in the force-transfer mechanism, an effective decrease in friction at the contact point of the material with the anvil, a difference in thermal-expansion coefficients of the material and the anvil, etc.

This can be illustrated by using the mechanical model in Fig. 3a. The resistance-load dependence in Fig. 3b was obtained by using this model. Almost all of the effects described in Refs. 1–9 and those attributed to the pressure-caused metallization by the authors of this paper can be demonstrated by using this model.

With regard to the behavior of two-component mixtures,⁸ we point out that two discontinuities in the R(F) curve are often observed even in a one-component substance (see Ref. 6). Since the investigated effects occur at a certain, previously unknown layer thickness,⁷ it is unclear how the authors⁸ have separated the essential effects from the nonessential effects.

The results of our experiments show that the data presented in Refs. 1–9, as well as those in Refs. 10–13, are ambiguous.

¹L. F. Vereshchagin, E. N. Yakovlev, G. N. Stepanov, and B. V. Vinogradov, Pis'ma Zh. Eksp. Teor. Fiz. 16, 382 (1972) [JETP Lett. 16, 270 (1972)].

²L. F. Vereshchagin, E. N. Yakovlev, B. V. Vinogradov, V. P. Sakun, and G. N. Stepanov, Pis'ma Zh. Eksp. Teor. Fiz. 17, 422 (1973) [JETP Lett. 17, 301 (1973)].

³L. F. Vereshchagin, E. N. Yakovlev, B. V. Vinogradov, V. P. Sakun, and G. N. Stepanov, Pis'ma Zh.

- Eksp. Teor. Fiz. 20, 472 (1974) [JETP Lett. 20, 215 (1974)].
- ⁴L. F. Vereshchagin, E. N. Yakovlev, B. V. Vinogradov, and V. P. Sakun, Pis'ma Zh. Eksp. Teor. Fiz. 20, 540 (1974) [JETP Lett. 20, 246 (1974)].
- ⁵L. F. Vereshchagin, E. N. Yakovlev, B. V. Vinogradov, and Yu. A. Timofeev, Fiz. Tverd. Tela 18, 113 (1976) [Sov. Phys. Solid State 18, 63 (1976)].
- ⁶L. F. Vereshchagin, E. N. Yakovley, and Yu. A. Timofeey, Pis'ma Zh. Eksp. Teor. Fiz. 21, 190 (1975) [JETP Lett. 21, 85 (1975)].
- ⁷L. F. Vereshchagin, E. N. Yakovlev, and Yu. A. Timofeev, Pis'ma Zh. Eksp. Teor, Fiz. 21, 643 (1975) [JETP Lett. 21, 304 (1975)].
- 8E. N. Yakovlev, L. F. Vereshchagin, B. V. Vinogradov, and Yu. A. Timofeev, Pis'ma Zh. Tekh. Fiz. 2. 570 (1976) [Sov. Tech. Phys. Lett. 2, 223 (1976)].
- ⁹B. V. Vinogradov, L. F. Vereshchagin, E. N. Yakovlev, and Yu. A. Timofeev, Pis'ma Zh. Tekh. Fiz. 2, 964 (1976) [Sov. Tech. Phys. Lett 2, 378 (1976)].
- ¹⁰N. Kawai and A. Nishiyama, Proc. Japan Acad. 50, 72 (1974). ¹¹N. Kawai and A. Nishiyama, Proc. Japan Acad. 50, 634 (1974).
- ¹²N. Kawai, M. Togaya, O. Mishima, and B. L. Neindre, Proc. Japan Acad. 51, 627 (1975).
- ¹³N. Kawai, M. Togava, and O. Mishima, Proc. Japan Acad. 51, 630 (1975).
- ¹⁴D. A. Liberman, J. Phys. Chem. Solids **39**, 255 (1978).
- ¹⁵A. L. Ruoff, High Pressure Science and Technology, Vol. 1, Ed. K. D. Timmerhaus and M. S. Barber, 1977, p. 753.
- ¹⁶H. K. Mao and P. M. Bell, Carnegie Inst. Yearbook 75, 509 (1976).
- ¹⁷H. K. Mao and P. M. Bell. Science 200, 1145 (1978).

Translated by Eugene R. Heath.

Edited by S. J. Amoretty