Ultramicromanipulator – A New Attachment for Electron Microscopes

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In the course of the last few years there has been a tendency in electron microscopy to provoke changes on the specimen during observation. Heating of the object and some forms of deformations, e. g. introduction of strains [1], etc., were made possible by the use of special specimen holders; evaporations have been carried out inside the electron microscopy [2] in order to observe the phenomenon of epitaxial growth, and so on. In this paper we propose a general tool to manipulate the specimen because we hope that by the aid of such a device, many new techniques may be introduced in electron microscopy.

For this purpose we have devised an ultra-micro-manipulator functioning inside the electron microscope which allows continuous micromovements above or under the object plane, in order to introduce external influences on the specimen in a chosen microdomain of the field of view. These micromovements must be roughly comparable to the resolution of a given electron microscope for the smallest scale unit of the external control device. In searching for such movements, two phenomena proved to be successful, namely:

1. The change in length of some flat springs of very high YOUNG modulus under deformation, the resulting micromovements being perpendicular to the direction of the deforming force. The movements cover the whole range between a few Ångström units and a few microns but they are not a linear function of the acting force.

2. The expansions and contractions of piezoelectric ceramics under the effect of electrostatic fields. This was investigated earlier by RAMSAY and MUGRIDGE [3] as a method of generating ultrafine movements though not for the purpose with which we are concerned here. In this case the range covered extends from a few Ångström units to a few microns and displacements are proportional to the voltage applied. The phenomenon is not free from hysteresis. This, however, can be minimized by a suitable prepolarization of the ceramic.

We want to present a brief account of a device based on the second principle.

A single element of the manipulator is composed of a disc of a piezoelectric ceramic, barium titanate, 3 mm in height and 6 mm in diameter. Three elements, the axes of which are orthogonal, form a unit for general micromovements (Fig. 1). Circular copper plates of 0.5 mm thickness serve as electrodes, cemented by a

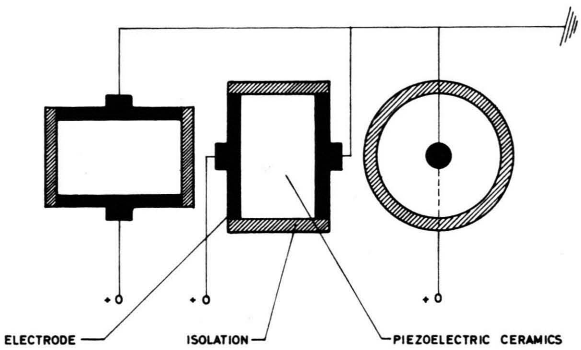

Fig. 1. Discs of piezoelectric ceramics arranged to give a general micro-displacement under the effect of electrostatic fields.

conducting Araldite compound to the two bases of the disc while a composition of Araldite and Thiokol, heat-polimerized on the surface, assures electric insulation, being elastic enough to follow, without rupture, the maximum expansion of the disc. The piezoelectric strain constant of the barium titanate compound was 1.9×10^{-10} Coul/Newton, and a displacement of about 2 μm could be obtained for a voltage of 10 kV. The discs were prepolarized, according to the cited work of RAMSAY and MUGRIDGE, by a field of 20 kV/cm, applied axially for one hour, at a temperature slightly above the CURIE point, which is 120 °C. By this polarization, displacements will vary linearly with the voltage applied. Recently we tried to apply the so called "non-modified" lead-zirconate-titanate ceramics described by CRAWFORD [4], because they have a strain amplitude per unit field greater by about 50% than that of the former material. Our ceramics are thickness expanders and the micromovements are solely axial in direction. In the case of barium titanate and when the prepolarizing field had the same polarity as the field throughout the manipulating work, only a very small hysteresis was present. In case of the lead-zirconate-titanate ceramics we have had great difficulties in prepolarizing because of a much higher CURIE point. However, as micromanipulation is in fact a process under visual observation, hysteresis does not disturb in most cases, that is, where there is no need of a strictly calibrated micromovement.

To obtain a general three dimensional movement, three identical piezoelectric elements are used in an assembly shown by Fig. 2. Each disc is carrying the next one, that is, a disc can be moved by its own driving voltage or be carried by a preceding disc in another direction. Each element has independent electrical leads and is driven by a separate high tension source. Small nests of a plastic material serve to establish mechanical connection between the elements and

[1] H. G. F. WILSDORF, L. CINQUINA, and C. J. VARKER, Proc. 4th Intern. Conf. on Electron Microscopy, Berlin 1958, p. 559 [1960].

[2] G. A. BASSETT, Proc. European Reg. Conf. on Electron Microscopy, Delft 1960, p. 270 [1961].

[3] J. V. RAMSAY and E. G. V. MUGRIDGE, J. Sci. Instr. **39**, 636 [1962].

[4] A. E. CRAWFORD, Brit. J. Appl. Phys. **12**, 529 [1961].

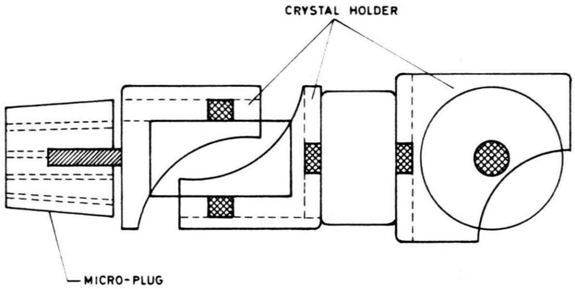

Fig. 2. Piezoelectric unit composed of three piezoelectric condensers.

at the same time assure both electrical insulation between discs and orthogonality of disc axes. The last element carries a minute holder (manipulatig arm) for the microtool which is to be brought in the field of view to interact with the specimen. The tool holder is electrically isolated from the piezoelectric unit. The movement of each element can be operated independently, or two or three simultaneously, by independent controlling organs. The unit composed of three elements is attached to a cap in which are located the extremities of the electrical leads. By this cap, the whole unit is simply plugged in a slender long cylinder which contains electrical leads conducting from piezoelectric unit to the high tension sources (Fig. 3 *). It is thus rigidly bound to the piezoelectric unit and therefore by carrying out mechanical manipulations on this cylinder, a preliminary adjustment of the position of the piezoelectric unit can be obtained. In Fig. 3 we see also the three-element piezoelectric unit with micro-plug and the external manipulating device. This latter allows the advancement of the piezoelectric unit towards the optical axis without rotation, and two other translations, one paralel to the specimen plane, one perpendicular to it. Furthermore, a system of gear and pinion serves to rotate the unit around an axis perpendicular to the electron beam. The whole piezoelectric unit is inside the electron microscope (Fig. 4). It is contained wholly in the cylindrical hole cut through the specimen stage, perpendicularly to the optical axis. This cavity acts at the same time as electrical shielding surrounding the piezoelectric unit.

In a first approach we used a Philips EM 100 electron microscope for demonstration and testing, in which alterations could be made without a possible damage to a more costly instrument. Obviously there is a need of adaptation for each type of electron microscope. The form of the specimen holder of the Philips electron microscopes is propitious for coming near the specimen itself, but even this specimen holder had to be slightly modified. Manipulations on the specimen can be caried out from above or beneath the specimen plane. However, as manipulation of the specimen may consist in influencing the beam, and because of the presence of the objectiv diaphragm under the plane of the specimen, it is more advisable to manipulate from above.

The microtools may be micro-needles, micro-capillaries, micro-electrodes, micro-diaphragms, micro-containers, micro-sources of evaporation and other means and materials being able to provoke changes on the specimen. The expression of "manipulating arm" is meant in a broad sense: it may be represented by every mean permitting the introduction of materials other than the specimen in the interior of the electron microscope. For example, the manipulating arm may be designed so as to permit the introduction of small quantities of gases or solid materials, for gaseous-solid or solid-solid reactions and so on.

The testing of micromovements was carried out (previous to the construction of the model) with the MOIRÉ-techniques of optical gratings. Once at attachment is made however, testing becomes particularly easy by observing movements in the electron microscope. Fig. 5 shows a demonstration of these testings. Here a needle of a ZnO crystal is moved by the manipulator relative to a hole in a formvar film in the specimen holder.

As to the applications of the tool, we have already devised numerous new techniques. Two of these, under elaboration presently, are an ultramicro indentation technique in which a monocrystalline specimen is attacked by a ZnO needle in order to provoke the generation of dislocations under observation, and the creation of MOIRÉ-figures under observation so that a crystal is superposed by the manipulator on the crystalline specimen in a rotating specimen holder.

The results of these techniques together with further new methods will be described elsewhere.

* Fig. 3—5 on p. 1728 a.

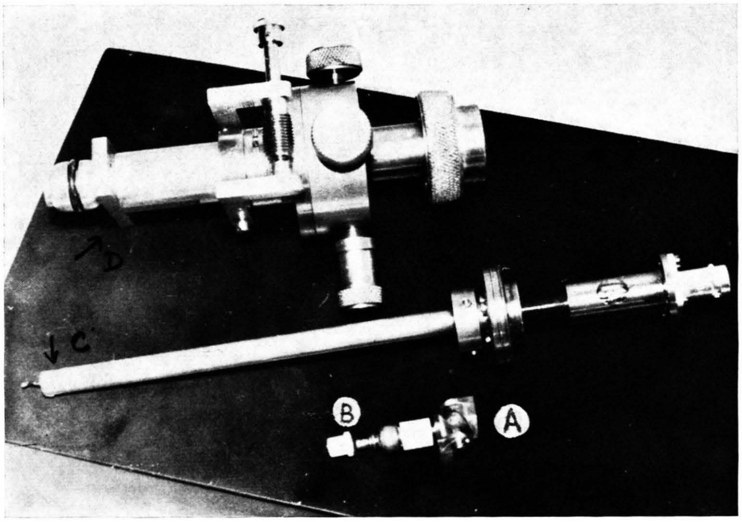

Fig. 3. Showing the three-element piezoelectric unit (A); micro-plug (B); the slender cylinder (C) to which the unit is attached and the external manipulating device of the latter (D).

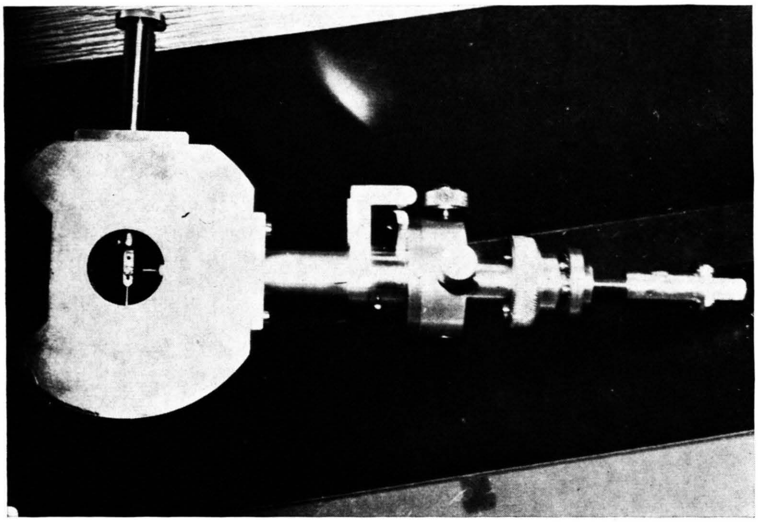

Fig. 4. Showing the ultramicromanipulator in the specimen stage of a Philips EM-100 electron microscope.

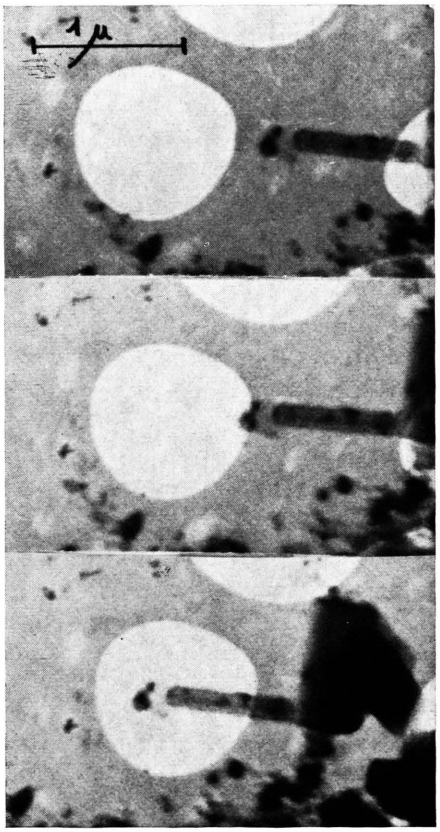

Fig. 5. Demonstrating micromovements produced by one member of the piezoelectric unit in which a ZnO needle is moving relative to a hole in a formvar film.

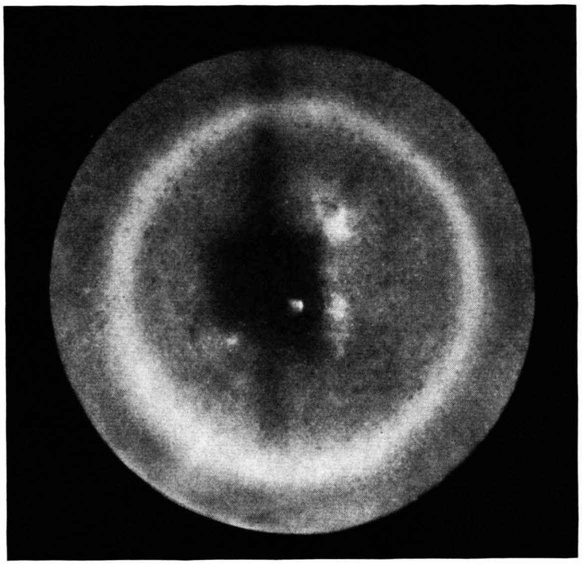

Abb. 1. Ringreflex, Unterlagentemperatur T_u=435 °C.

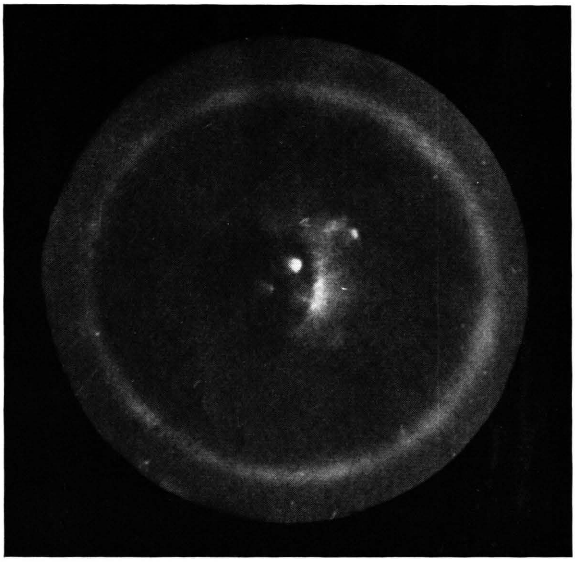

Abb. 2. Ringreflex mit Struktur, T_u=480 °C.

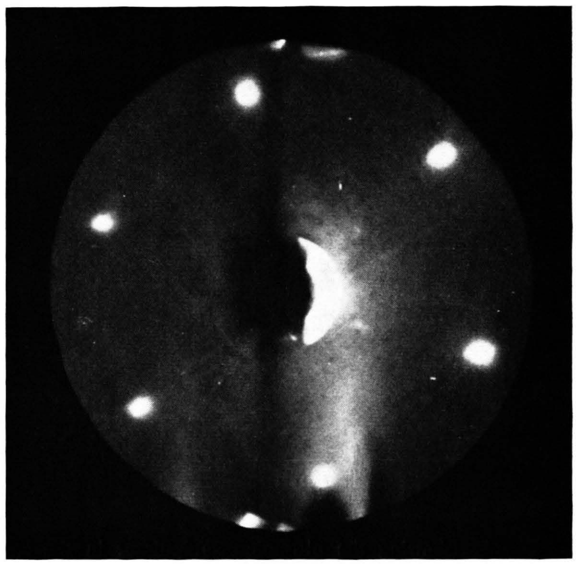

Abb. 3. Punktreflexe, T_u=550 °C.