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Fiber microelectrodes for electrophysiological recordings

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Methods for the fabrication of tungsten-glass and platinum-rhodium-quartz fiber microelectrodes and of fiber pipettes are described and the electrical and mechanical properties of fiber electrodes are discussed. These properties (minimal tissue damage, good single unit isolation and temporal stability) make them particularly suited for multielectrode recordings from the central nervous system.

Introduction

Metal microelectrodes are widely used in neurophysiology, and a large variety of fabrication techniques has been reported (Grundfest et al., 1950; Hubel, 1957; Gesteland et al., 1959; Wolbarsht et al., 1960; Marg, 1964; Guld, 1964; Frank and Becker, 1964; Baldwin et al., 1965; Müller and Vatter, 1969; Merrill and Ainsworth, 1972; Levick, 1972; Garoutte and Lie, 1972; Merrill, 1974; Braga et al., 1977). In some experimental situations, however, these electrodes have drawbacks: (a) exact control not only of the tip size, but of the tip geometry is difficult to achieve with conventional microelectrode fabrication techniques. Exact control of tip geometry is desirable if many electrodes with identical electrical characteristics are required, or if the tip geometry is to be optimized for a particular recording situation; (b) the diameter of the shank cone of these electrodes increases with penetration depth, and it can become large enough to cause serious displacement or rupture of superior tissue if large penetration depths are required; (c) the shaft diameter of conventional microelectrodes, generally, is large (up to 1 mm). It is, therefore, not possible to use such electrodes in closely spaced parallel arrays for multi-unit recordings.

In order to overcome these drawbacks, we sought to reduce the gross dimensions of the microelectrodes close to the limits of their mechanical stability and to develop a tip fabrication technique that permits exact and reproducible control of tip geometries. This is particularly important when arrays of many electrodes are used (Reitboeck et al., 1981; Reitboeck, 1983). We began experimenting with fiber

microelectrodes in 1972 and the early tungsten-glass fiber electrodes feasible for electrophysiological applications were used for recordings from the optic tract, geniculatum, superior colliculus, and visual cortex of the cat (Kozak and Reitboeck, 1974). Since that time, fiber electrodes have been used extensively in our laboratory (Habbel and Eckhorn, 1981; Krause and Eckhorn, 1981; Lohmann and Eckhorn, 1981; Reitboeck et al., 1981; Schneider and Rohde, 1981; Reitboeck and Werner, 1983; Reitboeck, 1983; Schneider et al., 1983) and also by other researchers (Krüger, 1982). In the following, the mechanical and electrical properties of fiber electrodes will be discussed, and various types of glass–metal fiber electrodes and fiber pipettes that have been developed in our laboratory will be described.

Fiber electrode fabrication

The starting material for the fiber electrodes are thin glass or quartz fibers with a metal core of matching thermal expansion coefficient. Suitable dimensions are 20–100 μm o.d. of the glass or quartz mantle, and a diameter of 12 μm or less for the metal core. One method for the fabrication of such fibers is based on technologies developed originally for optical glass fibers, as used in electro-optical communication systems. For electrophysiological applications, however, the central glass core of the optical fibers is replaced by a metal core during the fabrication process.

If optical fiber fabrication techniques are used, the metal core has to have a higher melting point than the glass mantle. Glass–tungsten fibers are made by this process. Companies that fabricate optical fibers or so-called "multi-lead-substrates" (glass plates with tungsten fiber inclusions as used in electron-beam printing tubes) can supply glass tungsten fibers on special order *. Fibers with other core metals (gold, iron, nickel, platinum, iridium, and their alloys) are easiest made by a process originally described by Taylor (1924).

Tungsten–glass fiber electrodes

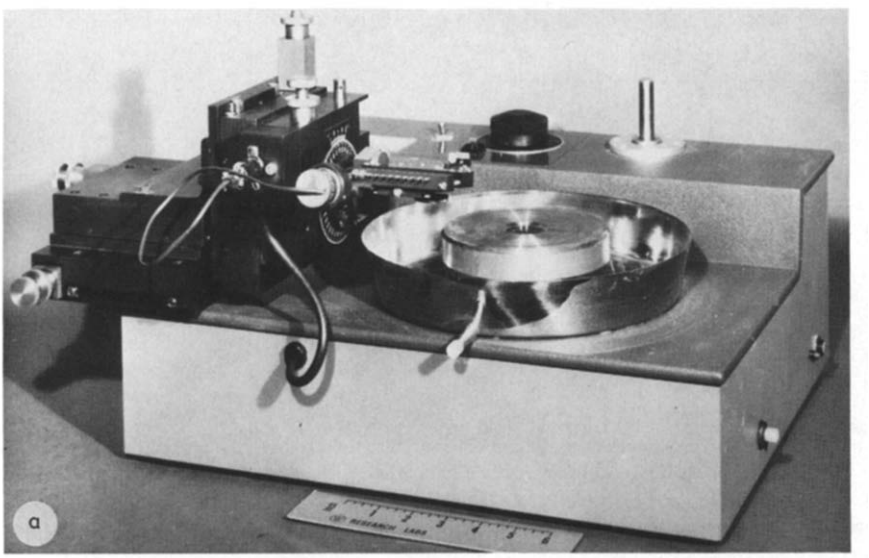
The fibers we are using consist of a 12 μm tungsten core, surrounded by a Nonex ** glass mantle of 100 μm o.d. For these fibers the tip fabrication technique that gives best results is to grind a conical tip onto the fiber with a fine grade diamond grinding wheel (8 cm diameter, 5–20 rps) or with a copper clad steel disk (15–20 cm diameter, 1–2 rps), covered with diamond grinding paste. The electrode itself is held with wax in a glass pipette whose tip i.d. is about the size of the fiber o.d., or in stainless-steel hypodermic tubing with suitably small i.d.. The tip geometry can be controlled by setting the angle between the electrode axis and the grinding disk. The electrode holder (attached to a micro-drive) is adjusted such that the end of the fiber slightly bends when it touches the grinding disk; this generates

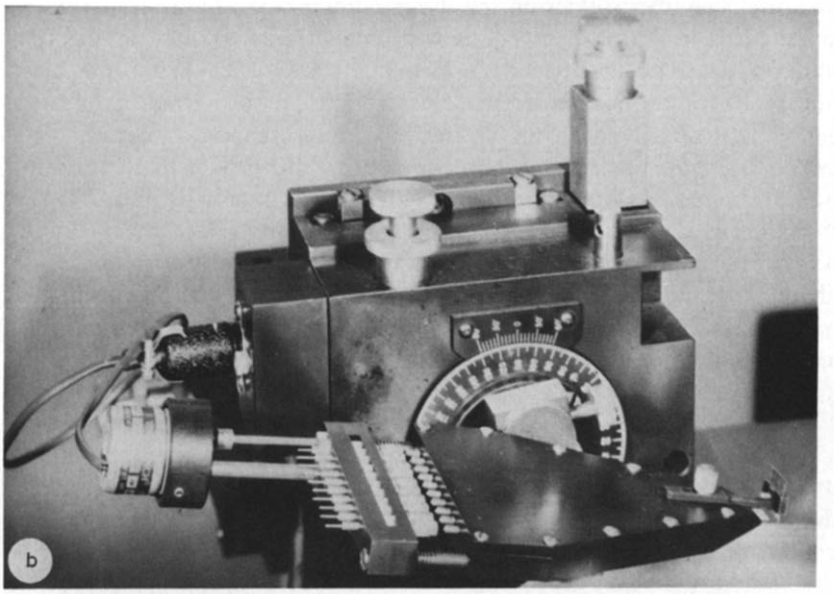
* e.g. Corning Glass Works, Electronic Materials Department, Corning, NY 14830, U.S.A.

** Other suitable glasses are Osram 362 a, and Schott 1646 III.

sufficient pressure. The fiber rotates during the grinding process in order to obtain a perfectly centered conical tip. The required grinding time depends on the fiber diameter and on the type and speed of grinding wheel used. With fine grade diamond wheels, 10 rps and 3.5 cm electrode eccentricity, typical grinding times are less than 1 min for a single conical tip. Overgriding cannot happen; when the tip is finished, there is no pressure any more between fiber and disk, and the grinding stops by itself. If desired, the tip area can be monitored electrically by measuring the tip impedance during grinding.

A machine for the simultaneous grinding of 10 fibers is shown in Fig. 1a,b, and a schematic diagram of the grinding process is shown in Fig. 1c. With this technique, fiber microelectrodes with relatively low impedances (150 kΩ–1 MΩ at 1 kHz) can be fabricated, which are suitable for recordings from large cortical neurons. The grinding process generates microgrooves in the metallic tip, such that the effective tip surface is much larger, and the tip impedance considerably smaller than in electrolytically etched tips of the same overall tip volume. The mechanical stability of the electrodes is very high: a 100 μm o.d. fiber electrode can penetrate even the hardened dura in chronic preparations. At an axial force of 10 p (0.35 oz.) the free buckling length (outside the tissue) is 8.5 mm (see Appendix). In tissue, the fibers can penetrate several times that deep without significant axial deviation. In order to minimize tissue disturbances at the recording site, the electrode tip angle should be as small as possible. In fibers with a 12 μm core, however, a small tip angle would result in an exposed metal area that is too large for good single unit isolation. This dilemma can be avoided with double-conical tips. Fig. 2 shows tungsten–glass fiber microelectrodes with a conical and a double-conical tip.





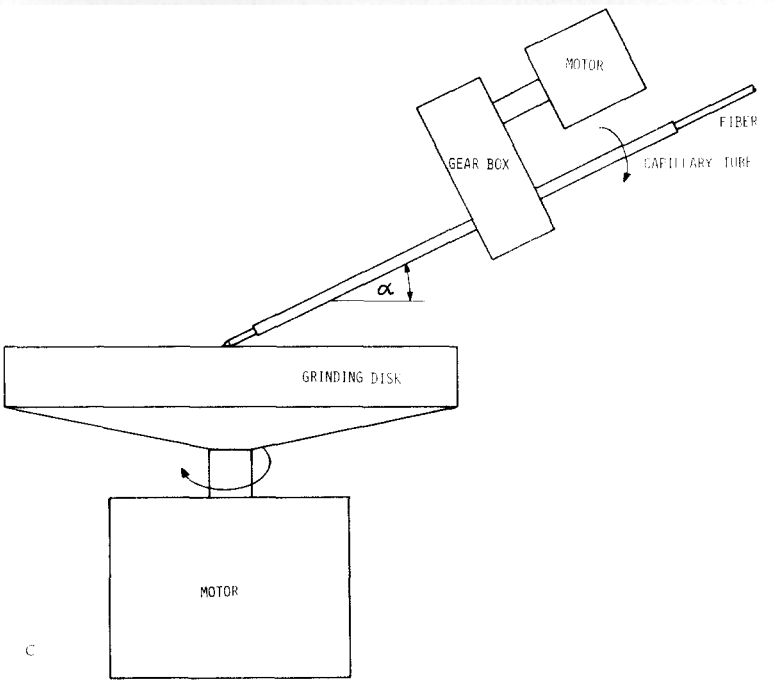


Fig. 1. a: machine for the simultaneous grinding of 10 fiber microelectrodes. The desired tip angle can be set on the scale shown in b. Exact positioning of the fibers relative to the grinding disc is possible via the x- and y-controls of a micro-drive. The fibers rotate inside stainless-steel capillaries (120–140 μm) i.d. for guidance and in order to increase the grinding force. c. schematic diagram of grinding process for a single fiber.

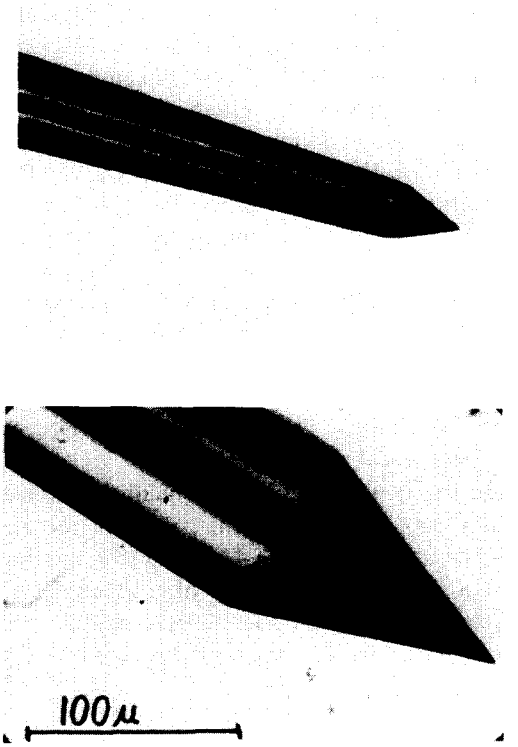


Fig. 2. Tungsten-glass fiber microelectrodes with a double-conical (upper part) and a conical tip. Note that the tungsten core diameter does not taper.

Platinum-rhodium-quartz fiber electrodes

Since tungsten glass fibers with core diameters of less than 10 μm are not available, we used Taylor wires (Taylor, 1931) for the fabrication of fiber electrodes with very small tips. This technique involves the drawing of a tip onto the fiber in a similar way as it is done with glass tubes in a conventional pipette puller. Since the fiber contains a metallic core, the technique requires that the temperature of the heating element exceeds the melting point of the core. The difference in the melting properties of metals and glasses make this process possible: in metals, the transition from the solid to the molten state is rather abrupt, whereas in glasses the transition is over a wide temperature range with changing viscosity. During the drawing of the tip, the viscous glass cone helps to contain the molten core. Since it is on this difference in the melting properties of glasses and metals that the fabrication of Taylor wires (Taylor, 1924; 1931) is based, Taylor wires make an ideal starting material for this tip fabrication technique.

A process for the fabrication of continuous strands of Taylor wires has been

described by Nixdorf (1967). A fiber drawing machine, modified by our mechanics workshop is available in our laboratory; its mechanical design is simple, so that any laboratory with access to a good mechanical and electrical workshop can have one built *. A short summary of the fiber drawing process is as follows: the starting material is a tube of quartz glass with an o.d. of a few millimeters. A piece of wire of the desired core material, e.g. platinum–rhodium ** is placed inside this tube. The desired ratio of the glass/metal diameters in the cross-section of the fiber is determined by the same ratio in the starting materials. One end of the quartz glass tube is subsequently sealed in a hydrogen flame, and the open side of the tube is connected to a high-vacuum pump. During evacuation the assembly is heated to 1000°C for complete degasification. The other end of the rod is then sealed, and the rod is subsequently placed into the drawing machine. The drawing machine consists of a resistive heating element which can produce temperatures up to 2100°C (e.g. an iridium coil) through which the glass tube protrudes. The tube itself is moved in axial direction at a very slow speed by a mechanical drive assembly.

When current is turned on, and when the temperature of the heating element reaches the melting point of quartz glass (1700–2100°C), a thin stream of molten glass flows down, which solidifies quickly as soon as it is outside the heating element. This fiber is then attached to a rotating drum. The fiber diameter is determined by the ratio of the speed of the drum and the speed of the supply rod. The condition is that the volume of the material withdrawn must be equal to the volume of the material supplied:

$$\frac{D^2\pi}{4} v_s = \frac{d^2\pi}{4} v_d$$

where D = diameter of the quartz tube; d = fiber diameter; v_s = speed of the quartz tube in the supply assembly; and v_d = speed of the drum.

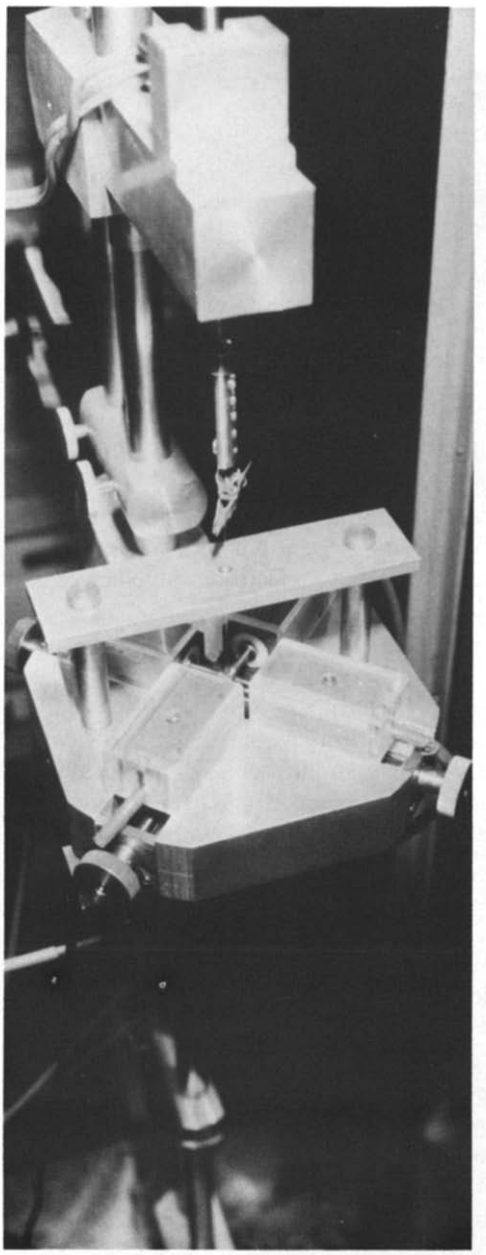
v_d and v_s are coupled electronically, so that fibers of uniform dimensions are produced. With this technique fibers with an o.d. of 200 μm to 20 μm and with core diameters from 50 μm down to about 2 μm can be fabricated.

In order to make electrodes from these fibers, the same grinding process as described for the tunsgten electrodes can be used. For the fabrication of electrodes with very fine tips we have developed a machine which makes tips down to the submicron range. This machine operates in a fashion similar to a conventional micropipette puller. Since it has to melt quartz glass the temperature has to be much higher, however. A electric arc in a quadrupole configuration for centric symmetric heat distribution is used to heat the fiber. Since the time-course of the pulling force

* Taylor wires are used also in fiber-reinforced materials. Companies that fabricate such materials might be able to supply Taylor wires on special order.

** Platinum–rhodium is our preferred core material, since platinum–rhodium wires as used in thermocouples are suitable and readily available. Its hardness, its resistance against oxidation and corrosion, and its good tissue compatibility make platinum–rhodium well-suited for electrophysiological applications. If other core materials are used, a glass type has to be selected that reaches a viscosity suitable for fiber drawing at a temperature that is higher than the melting point of the core.

has to be controlled exactly, we are using a moving coil assembly from a loudspeaker which is activated by a programmable ramp generator. The drawing chamber is also



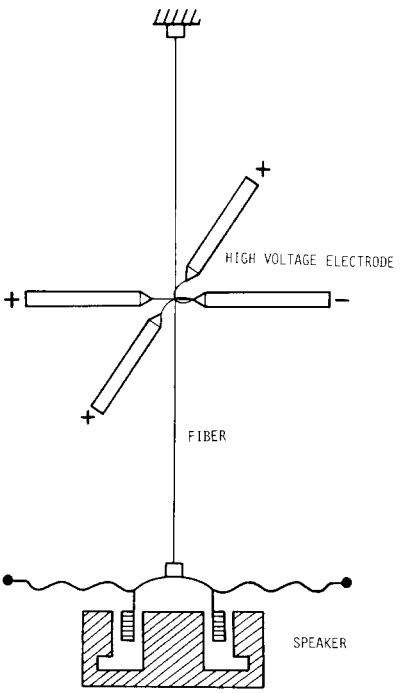


Fig. 3. a: electrode puller for quartz fiber electrodes. The fiber is heated by an electric arc in quadrupole configuration. b: schematic diagram of electrode puller function.

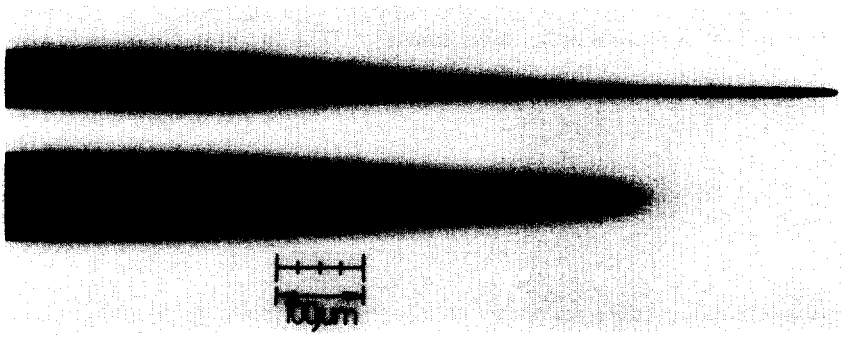


Fig. 4. Platinum-rhodium-quartz fiber microelectrodes with very fine tips. Note that the core conductor tapers toward the tip. In the electrode shown in the upper part, the tip core diameter is approximately 1 μm.

shielded from external air turbulances in order to avoid microscopic bending of the very tip of the electrode. The fiber electrode puller is shown in Fig. 3. With this technique we have been able to fabricate microelectrodes from fibers ranging from 100 to 20 μm o.d. with perfectly straight tips down to the submicron range (Fig. 4). With additional grinding of the tips, as described above, electrode impedances can be optimized for a particular neurophysiological application.

Fiber pipettes

The drawing and tip fabrication process described above has been used also in order to make fiber pipettes. In this case, no metallic core is put inside the quartz-glass tube when the fibers are drawn; if a thin quartz-glass rod is put inside the quartz-glass tube *, subsequent filling of the fiber electrodes with an electrolyte is greatly facilitated.

Electrode contacting

Contacting of the electrodes can be done in several ways: for fiber microelectrodes with relatively large core diameters (10 μm and more), contacting is done by first sleeving a thin copper tube (with an i.d. slightly larger than the o.d. of the fiber) over the fiber electrode. The quartz mantle is then broken so that the metallic core is exposed, and the copper tube is sleeved over the exposed wire and clinched with a pair of tweezers. This results in a very good electrical contact between the copper tube and the metallic core of the fiber. For added mechanical stability, the copper tube is glued to the fiber with a droplet of Expoy. For very fine fibers (20–40 μm

* Suggested by R. Eckhorn; a similar technique is also used in conventional micropipettes.

o.d. and core diameters of 5 μm and less) this technique is not applicable any more. Such fibers are contacted by first etching off a short segment of the quartz mantle *. After washing and drying, contacting is done with a droplet of conducting silver paint.

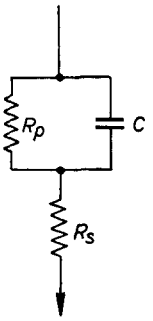


Fig. 5. Equivalent circuit for electrode tip impedance.

Electrical characteristics of ground fiber electrodes

The tip impedance of metal electrodes can be approximated by the equivalent circuit of Fig. 5. The capacitance C, generally, is frequency-dependent. This, and other impedance variations due to changes in the electric double-layer at the metal/electrolyte interface (Frank and Becker, 1964; Epelboin and Keddam, 1970) make it difficult to give exact values for C, R_s and R_p. Tungsten electrodes have the additional problem of surface oxidation, which can greatly change the electrode impedance. The impedance of freshly ground tungsten–glass fiber electrodes was measured at 15 frequencies within the frequency range of 100–10,000 Hz. Reproducibility of the impedance values within $\pm 8\%$ was obtained by frequent ultrasonic cleaning of the electrode while immersed in the electrolyte. Averages of several runs resulted in the following values:

$c = 2.2\ \mathrm{pF}/\mu\mathrm{m}^2$, $r_p = 285\ \mathrm{M}\Omega \cdot \mu\mathrm{m}^2$, $r_s = 27\ \mathrm{M}\Omega \cdot \mu\mathrm{m}^2$

with $C = c \cdot A$, $R_s = r_s/A$, $R_p = r_p/A$, A ... electrode tip area in $\mu\mathrm{m}^2$.

The characteristic tip capacitance (2.2 $\mathrm{pF}/\mu\mathrm{m}^2$) of this electrodes is considerably higher than the 0.2–1 $\mathrm{pF}/\mu\mathrm{m}^2$, typically measured on chemically etched tips (Robinson, 1968). This is probably due to microgrooves, caused by the grinding process. The high tip capacitance is responsible for the excellent electrical properties of ground fiber electrodes.

An example of extracellular spike potentials from pyramid cells in cat visual cortex, recorded with a fiber electrode, is shown in Fig. 6.

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* The etching can be done by immersion in hydrofluoric acid, or, better, in molten alkaline salts. We are using sodium carbonate ($Na_2CO_3 \cdot 10\ H_2O$, cryst.).

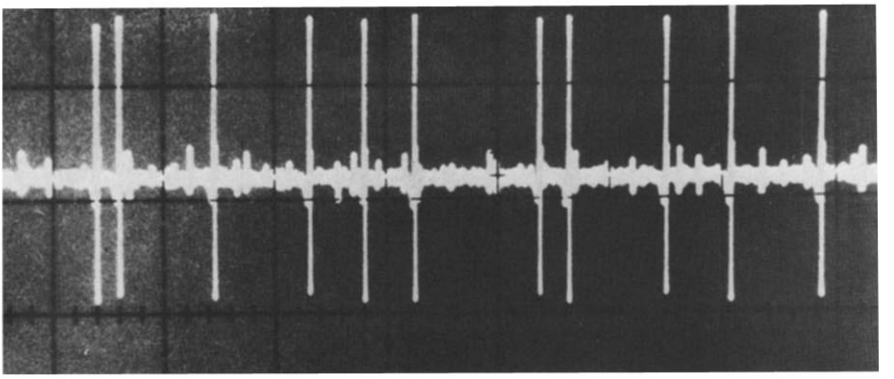

Fig. 6. Sequence of spikes recorded from pyramid cells in cat visual cortex (fiber microelectrode; exposed metal tip: 4 μm base diameter, 8 μm length).

Properties of fiber electrodes

The fiber electrodes have a number of advantages over conventional microelectrodes as well as over electrodes fabricated with photolithographic methods. These advantages are the following.

(1) The shaft of the electrodes, over the whole penetration depth, is cylindrical and very thin. Electrodes with fiber diameters ranging from 100 to 30 μm have been made, and even 20 μm is possible. Due to their small dimensions, the electrodes cause minimal tissue damage. The electrodes have no cutting edges, as many silicon-substrate electrodes do.

(2) The mechanical stability of the fibers is very high, perhaps close to the limit of what is possible at these dimensions with any material at this time. Fibers with 30 μm o.d. at an axial force of 1.5 p (0.05 oz., which is much more than required for penetration of the pia, when the dura is removed) have a buckling length of 2 mm. As long as this unguided fiber length outside the tissue is not exceeded, 30 μm-fibers can penetrate tissue to depths of up to 5 mm. For fiber electrodes of 100 μm o.d. and 6 p (0.21 oz.) axial force (more than required to penetrate the hardened dura in a chronic preparation) the buckling length is more than 10 mm. At an axial force of 20 p (0.71 oz.), the buckling length is still 6 mm. In tissue, the penetration depth of 100 μm o.d. electrodes can be 20 mm and more.

(3) Due to the geometrical shape of the fibers, tissue is displaced radially during penetration, with little axial tissue compression. There is little or no re-adjustment of tissue after insertion, which might be one of the reasons why injury potentials are hardly ever seen after electrode movement has been stopped.

(4) The geometrical shape of the electrode tip can be made exactly and reproducably according to specifications, as long as it is a tip geometry that is grindable. Conical tips and elliptical cuts can be reproduced with high accuracy.

(5) Additional electrolytic plating of the platinum–rhodium tips is unprob-

lematic. We have fabricated fiber electrodes with gold-, iron-, and silver/silver–chloride-plated tips. Iron-plated tips can be used for electrolytic staining of the recording site. The silver–chloride-tipped fiber electrodes have excellent low frequency responses and DC stability.

(6) Microgrooves, caused by the grinding process, increase the effective tip area at a given tip volume. This results in a tip capacitance of more than 2 $pF/\mu m^2$, which is considerably higher than the tip capacitance of etched tips. The high tip capacitance seems to be responsible for the excellent signal-to-noise ratio and single-unit isolation of the electrodes.

(7) Fiber electrodes are well-suited for multi-electrode arrays. In contrast to silicon-substrate electrodes (Wise and Angell, 1969; Wise and Starr, 1969) movement control of individual electrodes is possible (Reitböck and Werner, 1976, 1983; Reitboeck et al., 1981; Reitboeck, 1983).

Appendix

Glass–metal fibers: mechanical considerations

Is glass-metal fibers are used in a multielectrode configuration, data on the mechanical strength of the fibers are required for the design of the electrode manipulator. In the following, the permissible axial force for various fiber lengths (at 3 load conditions) will be calculated.

Due to the small diameter/length ratio of the fibers, the stress condition that is most likely to lead to fiber breakage in typical tissue penetration situations is not compression, but buckling. Buckling is a differential instability, that occurs in slender structures when no, or insufficient lateral counterforces are present. It is, therefore, unlikely to happen at electrode sections that have penetrated the tissue already. The axial force at which buckling occurs depends not only on the fiber diameter and on the free fiber length, but also on the boundary conditions, i.e. whether the fiber is guided or not. For electrodes, the following 3 cases are of interest.

Case I: the fiber section is unguided at both ends. Electrode extending from a wide guide tube, before penetration; Fig. 7a.

$$F_B = \frac{\pi^2 EI}{l^2}.$$

where F_B = buckling force (axial); E = module of elasticity (Young's module); $E = 7.3 \cdot 10^5$ kp cm^{-2} for fused quartz and about the same for glass; I = moment of inertia of the fiber cross-section; l = length of unguided fiber section.

Case II: the fiber is guided at one end. Electrode extending from close-fitting guide tube or glued into glass pipette holder, before penetration; Fig. 7b.

$$F_B = \frac{2.046\pi^2 EI}{l^2}$$

Case III: the fiber is guided at both ends. Electrode extending from holder or

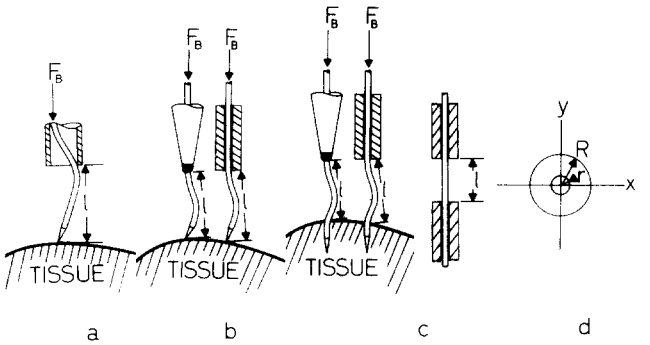

Fig. 7. Fiber load conditions in various electrophysiological recording situations (see text).

close-fitting guide tube after penetration, or electrode section between close-fitting guide tubes as in parts of the electrode manipulator mechanism (Reitboeck et al., 1981), Fig. 7c.

$$F_B = \frac{4\pi^2 EI}{l^2}.$$

The axial moment of inertia of the electrode cross-section (Fig. 7d) is for the fiber capillaries:

$$I_x = I_y = \frac{\pi(R^4 - r^4)}{4}.$$

For $R \geqslant 3r$ $I_x = I_y \approx \frac{\pi R^4}{4}$, i.e. the core contribution can be neglected (error

TABLE 1

BUCKLING FORCE F_{B2} IN PONDS (1 p ≈ 0.0353 oz.), FOR FIBER DIAMETERS OF 20–100 μm AND FREE FIBER LENGTHS OF 1–20 mm (LOAD CASE II)

Axial force, F_{B2}, at which buckling occurs for various fiber diameters and free fiber lengths for case II. For case I, $F_{B1} \approx 0.5\ F_{B2}$, and for case III, $F_{B3} \approx 2F_{B2}$.

l (mm)	d(μm)						
	20	30	40	50	60	80	100
1	1.16	5.87	18.55	45.29	93.91	–	–
2	0.29	1.47	4.64	11.32	23.48	74.20	–
3	0.13	0.65	2.06	5.03	10.43	32.98	80.51
4	–	0.37	1.16	2.83	5.87	18.55	45.29
5	–	0.23	0.74	1.81	3.76	11.87	28.98
6	–	0.16	0.52	1.26	2.61	8.24	20.13
8	–	–	0.29	0.71	1.47	4.64	11.32
10	–	–	0.19	0.45	0.94	2.97	7.25
20	–	–	–	–	0.23	0.74	1.81

< 1%). In this case the buckling length of both, fibers with metal core and of capillaries of equal o.d. is about the same.

The required axial force depends on the tip geometry of the electrode, on the fiber diameter, and on the tissue to be penetrated. If the dura mater must be penetrated, the required force is about 5–10 p. According to Table I, the permissible free fiber length is 10–8 mm (with a safety margin) for fibers of 100 μm o.d. If the dura and pia have been removed in a cortical preparation, the penetration force can be as low as 0.5 p. In this case, fiber electrodes as thin as 20 or 30 μm can be used at free fiber lengths of 1–3 mm.

Acknowledgements

Many improvements on the fiber electrodes resulted from their use in neurophysiological experiments in collaboration with Dr. G. Werner, University of Pittsburgh (1970–1975), Dr. W.M. Kozak, Carnegie-Mellon University (1972–1973), and Dr. R. Eckhorn in our Department. The tip drawing machine shown in Fig. 3 was designed by Mr. U. Thomas, who also made the electrodes shown in Fig. 4. The tip drawing machine and a machine for the fabrication of Taylor wires were built and modified respectively in our workshop by Mr. P. Muth, Mr. W. Gerber, and Mr. D. Mischke. Dr. Grünthaler, Battelle-Institut Frankfurt, gave us valuable advice on the fabrication of Taylor wires.

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