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Calibration of electrodiffusion friction probes using a voltage-step transient[*]

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Voltage-step transient experiments are used to calibrate electrodiffusion (ED) friction probes. The approach is demonstrated on the probes in high-speed rotating impellers. This calibration has shown that shear rates as high as $10^6\,\mathrm{s}^{-1}$ can be measured reliably using 0.5 mm Pt working electrodes in ferro/ferricyanide aqueous solutions. The complete transient calibration experiment provides sufficient information about the dynamics of ED friction probes.

Keywords: *friction probes*, *electrodiffusion*, *shear rates*, *calibration*, *transients*

List of symbols

A	electrode area (m^2)
a	Cottrell coefficient ($\mathrm{A\,s}^{1/2}$)
b	parameter, Equation 3(b)
c	depolarizer concentration ($\mathrm{mol\,m}^{-3}$)
D	diffusion coefficient of depolarizer ($\mathrm{m}^2\,\mathrm{s}^{-1}$)
D_∞	D at infinite dilution ($\mathrm{m}^2\,\mathrm{s}^{-1}$)
F	Faraday constant (96 485 $\mathrm{C\,mol}^{-1}$)
H^*	complex impedance, Equation 7 (A s)
I	limiting diffusion current (A)
K	parameter, Equation 1(b) ($\mathrm{A\,s}^{1/3}$)
k	shape factor
L	electrode length (m)
n	number of electrons involved in reaction of one mol (mol^{-1})
Pe	Péclet number ($Pe = L^2\gamma/D$)
R	radius of circular electrode (m)
R	ohmic resistance (Ω)
t	time (s)
t_0	characteristic time, Equation 3(a) (s)
T	temperature (K)
U	voltage (V)
W	electrode width (m)

Greek letters

γ	shear rate (s^{-1})
η	dynamic viscosity (Pa s)
σ	shear stress (Pa)
ϕ	electrode diameter (m)
ω	angular frequency ($\mathrm{rad\,s}^{-1}$)

Subscripts

C	Cottrell asymptote of transient
L	Leveque asymptote
exp	experimental

Superscripts

b	bulk
w	electrode surface
*	complex impedance of a fluctuating quantity

1. Introduction

Shear stress exerted by a flowing liquid on a solid boundary is an important parameter in equipment for handling microdispersed or biological liquids. The electrochemical method [1, 2], also known as the limiting diffusion current [3] or electrodiffusion (ED) technique [4, 5], is a reliable way of measuring wall shear stresses or shear rates, as both quantities can be simply related for a liquid with given viscosity, $\eta = \sigma/\gamma$.

An ED friction probe is an electrochemical cell which consists of a large auxiliary electrode and a small working electrode submersed in a flowing electrolyte solution. The probe should be designed and operated in such a way that all transport resistances outside the region of the working electrode are negligible. For this reason, the theory of electrodiffusion probes deals exclusively with transport processes inside the diffusion layer, adjacent to the working electrode and, when necessary, with the charge transfer, $\mathrm{Ox} + n\mathrm{e}^- = \mathrm{Red}$, across its surface. If the voltage, U, of an ED cell is sufficiently high to suppress the surface concentration of the working depolarizer to a negligibly small fraction of the bulk concentration, the current then becomes almost independent of U over a certain region (plateau) and reaches its limiting, diffusion controlled, value [1–7].

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2. Theory

2.1. *The Leveque formula and steady-state calibration*

The limiting diffusion current, I, can be calculated using the theory of convective diffusion for a single species [6], which is consumed by extremely fast reaction at the surface of the working electrode. If the Péclet number is so high that the effects of longitudinal [8] and transverse [9] diffusion can be neglected, the equation of convective diffusion under steady state can be easily solved [1–7]. The resulting Leveque formula predicts the limiting diffusion current for any working electrode geometry,

$$I_{\mathrm{L}}(\gamma) = K\gamma^{1/3} \tag{1a}$$

$$K = knc^{b}FAD^{2/3}L^{-1/3} \tag{1b}$$

where $L = R$, $A = \pi R^2$ and $k = 0.686\ 60$ for a circular electrode and $A = WL$, $k = 0.807\ 51$ for a rectangular electrode.

If all the basic properties were known, it would be possible to use the ED probe for absolute measurements of wall shear rates according to Equation 1. In practice, such circumstances are seldom met. Even if all simplifying assumptions are valid and the liquid composition is known exactly, it is difficult to make accurate estimates of the parameters A and D. The electrode area may be determined from a micrograph with accuracy $\pm 3\%$, assuming the surface is sufficiently smooth. The diffusivity depends in a complex manner on the solution composition and the concentration profiles in the diffusion layer [5, 10]. There are no reliable tabulated data for D, except at infinite dilution [6] which can differ from actual values by more than 20%. The concept of limiting diffusion currents, which are independent of applied voltage, U, over the plateau region, need not be valid. The actual I/γ curve can depend slightly (say $\pm 3\%$) on actually applied voltage.

In view of these uncertainties, ED probes should be calibrated under steady-flow conditions and carefully controlled voltage, using an appropriate hydraulic device to determine the calibration coefficient K in Equation 1. Checking the existence of a well-developed plateau by recording the I/U polarization curve is an obligatory part of such a calibration. The idea is attractive but it can only be applied if:

(i) a well-controlled steady viscometric flow can be generated in the setup used for the planned hydrodynamic experiments, for example, study of hydrodynamic instabilities in gravitational film flow [11] and Couette flow [12];

(ii) portable ED probes are used which can be calibrated in a specially designed device, an ED calibrator.

In other cases a complete calibration is either impractical or impracticable. Typical hindrances are that the ED probe is glued into a mechanical component, for example, an impeller blade [13, 14] or that the shear rates are too high, say $\gamma > 10^4\ \mathrm{s}^{-1}$, to be realized in a controllable viscometric flow.

In this paper, we recommend a way of *partially calibrating* (and checking) ED probes which cannot be subjected to a complete steady-state calibration procedure. The partial calibration consists of measuring the two electrochemical characteristics: (a) I/U polarization curves (at γ of the nonviscometric flow of planned experiments) and (b) voltage-step transient curves (no matter what γ).

2.2. *Cottrell asymptote and partial calibration*

A voltage-step transient experiment [15] starts from the zero-current state with the corresponding equilibrium voltage. At $t = 0$ a constant voltage U is applied which is high enough to guarantee the limiting diffusion regime under the ultimate steady-state condition. If the additional transport (ohmic and faradaic) resistances are negligible at any moment of the transient process, the applied voltage step causes a step change of the surface depolarizer concentration from $c^{\mathrm{w}} = c^{\mathrm{b}}$ to $c^{\mathrm{w}} = 0$ (or a negligible fraction of c^{b}). The transient current for a very short time after this step, $t < 0.5\,t_0$, can be calculated from the well-known Cottrell asymptote:

$$I_{\mathrm{c}}(t) = at^{-1/2} \tag{2a}$$

$$a = nc^{b}FA(D/\pi)^{1/2} \tag{2b}$$

The theory for the complete transient between Cottrell (2a, b) and Leveque (1a, b) asymptotes is known, both for rectangular [16] and circular [17] electrodes. We introduce the concept of the potentiostatic transient time t_0, defined as the time coordinate of the intersection of the asymptotes, $I_{\mathrm{c}}(t_0) = I_{\mathrm{L}}(\gamma)$, that is,

$$t_0 = (a/I_{\mathrm{L}})^2 = bD^{-1/3}(L/\gamma)^{2/3} \tag{3a}$$

$$b = 1/(k^2\pi) \tag{3b}$$

Here $b = 0.488\ 15$ for circular and $b = 0.675\ 21$ for rectangular electrodes. The theoretical transient currents can be described, with accuracy better than 1%, by the simple formula [16, 17]

$$\frac{I(t)}{I_{\mathrm{L}}} = \begin{cases} (t/t_0)^{-1/2} + (4/27)(t/t_0); & t/t_0 < 9/4 \\ 1 \hfill ; & t/t_0 > 9/4 \end{cases} \tag{4}$$

The partial calibration starts with the Cottrell coefficient, a, which is determined for the same ED friction probe and solution as used in the hydrodynamic measurements. The necessary transient experiment can be repeatedly done in the hydrodynamic setup or in the still solution, as a does not depend on γ. The formula

$$K/a = b^{-1/2}D^{1/6}L^{-1/3} \tag{5}$$

which follows from Equations 1(b), 2(b) and 3(b), makes it possible to estimate K from the known a and electrode geometry. Even with rough estimate of electrode area A, the depolarizer diffusivity can be estimated from known a with accuracy better than ±10%, which is satisfactory for the estimation of K according to Equation 5 with accuracy about ±2%.

The transient measurements in a region of very short times, that is, very high current densities, provide qualitative information about the extent of additional transport resistances. When the transient currents follow the Cottrell asymptote, $I \sim t^{-1/2}$, with reasonable accuracy, say ±2%, we can expect the steady currents of the same order of magnitude to follow the Leveque asymptote, $I \sim \gamma^{1/3}$, with the same accuracy. This semiquantitative information is extremely valuable for extrapolation of existing steady-flow calibration data into the region of very high shear rates. On the other hand, unusual transient behaviour (slower or faster decrease of $I(t)$ than $I \sim t^{-1/2}$) within the Cottrell range of transient times, $t < 0.5\, t_0$, give a warning that something is wrong: electrolytically poisoned electrodes if the currents are too low, leakage or roughness of the working electrode if the currents are too high.

Another useful feature, provided by the partial calibration, is the opportunity to correct K for unwanted changes of depolarizer concentration and diffusivity. Assuming the geometric parameters of the ED probe are unchanged, it is possible to use the equation

$$K_1/K_2 = (D_1/D_2)^{1/6}/(a_1/a_2) \qquad (6)$$

where 1 and 2 correspond to two solutions of similar composition at different temperatures and with different content of the same working depolarizer. The effect of absolute temperature, T, on diffusivity can be correlated with the viscosity data, $\eta_1 D_1/T_1 = \eta_2 D_2/T_2$.

For a series of embedded electrodes, the partial calibration can be used for determining the areas by comparing their Cottrell coefficients with that for a portable ED probe of similar size and known area A. Under the same experimental conditions (electrode material and roughness, polarizing voltage, temperature, and composition of the solution), a_{exp} are proportional to the electrode areas.

3. Experimental details

3.1. *Hydraulic devices, electrodes, solutions, data acquisition*

The experiments were carried out in a Couette-flow calibrator, in a hydraulic setup with a rotating disc and a pump impeller [13, 14, 18], and in a liquid film flowing down an oscillating plate [11, 24]. The Couette flow was generated by a flat cylinder of diameter 317 mm rotating in a cylindrical vessel of inside diameter 319.2 mm, (Fig. 1). The shear rate could be adjusted in the range 3–400 s^{-1}. Rectangular (0.11 mm × 0.99 mm) and circular (dia. 0.5 mm) electrodes were fabricated from platinum sheet and wire. The platinum was insulated by a polymeric film and glued by Epoxy resin into a stainless steel tube with an outside diameter of 5 mm and wall thickness 1 mm. The electrode was then polished with emery paper of grit size 15 μm. The probe was pushed through the perpendicular hole in the vessel wall and sealed by a rubber O-ring. The tube was connected with a large platinum auxiliary electrode and shielded the working electrode. These portable probes were mounted flush with the wetted wall of the calibrator or oscillating plate. Other electrodes made of insulated platinum wire with a diameter of 0.5 mm were firmly glued in the disc and impeller.

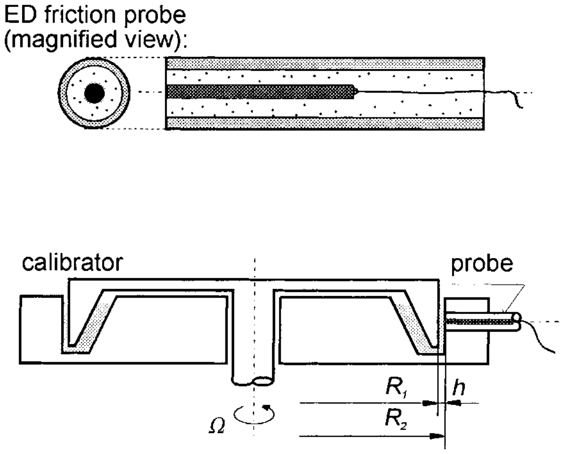

Fig. 1. Calibrator and a portable ED friction probe.

The liquid used was a 25 mol m^{-3} equimolar potassium ferro/ferricyanide aqueous solution with a 2% K_2SO_4 supporting electrolyte.

A special six-channel electrodiffusion analyser was used in the experiments. The main part of a channel consisted of two operational amplifiers, the first working as current follower and the second as amplifier. The analyser operated on six input ranges 1–10^5 μA. The polarizing voltage was controlled either manually or by a D/A converter of the PC twelve bit board DAS 16F. The voltage signal corresponding to the electrode current was recorded by an A/D converter of the same PC board. Software ED-WORK91 [25] was used for the signal processing to provide I/t (transient), I/U (polarization), and I/γ (calibration) curves and to analyse fluctuating wall shear rates in the several hydraulic units.

There are three critical points in measurement of the transient curves. The voltage step and current measurement must be carried out in an exact time sequence. This problem was handled by the software, which was based on our routines for the D/A and A/D conversions. In this way it is guaranteed that the command for A/D conversion follows the command for the voltage step with a known delay. The second problem is a rather large range of currents measured during the transient. The amplification of the ED analyser must match the highest current; in our case the current range was 1000 μA, whereas the steady state currents are by one to two orders lower. Therefore, it is necessary to adjust the offsets of the ED analyser and A/D converter precisely. The third problem is associated with the initial conditions. The depolarizer concentration at the electrode surface must be equal to the bulk concentration at $t < 0$, which implies that the current must be zero. In the case of the same material for the working and auxiliary electrodes, this condition is fulfilled by the imposition of zero voltage.

3.2. *An example of complete calibration*

A complete set of calibration data for a Pt circular probe with diameter 0.5 mm is shown in Figs 2–4. The polarization curves were measured in the regime of cathodic polarization, $-0.9\,\mathrm{V} < U < 0$, with a scan rate ($10\,\mathrm{mV\,s^{-1}}$) low enough to obtain steady values with no hysteresis effect. A broad plateau of limiting diffusion currents from −0.9 to −0.2 V indicates negligible ohmic and faradaic resistances for shear rates up to $330\,\mathrm{s^{-1}}$.

A fixed polarization voltage, $U = -0.8\,\mathrm{V}$, was imposed in all steady and transient experiments (Figs 3, 4 and Table 1). In Fig. 3 the current measured without additional resistance follows the theory, $\mathrm{dln}\,I/\mathrm{dln}\,\gamma = 1/3$ for $\gamma \in (30, 330\,\mathrm{s^{-1}})$. At higher shear rates, viscometric flow in the calibrator becomes unstable.

One thousand data in each transient run were recorded with sampling at 2 kHz. Therefore, the first point has a delay 0.5 ms after the voltage step. The maximum currents are equal to $353\,\mu\mathrm{A}$. This value is only slightly higher than that corresponding to the Cottrell asymptote (see Fig. 4). It can be concluded that neither additional transport resistance nor surface roughness are important at currents below $350\,\mu\mathrm{A}$. This current corresponds to a shear rate as high as $10^6\,\mathrm{s^{-1}}$, according to the extrapolated steady-state calibration curve. The first two rows in Table 1 confirm the suggested approach on a quantitative level. The Cottrell coefficients, a_{exp}, obtained by fitting the transient data by Equation 2(a) in the interval shorter than 0.02 s, were used for calculating the diffusivities, D, from Equation 2(b), and then the Leveque coefficients K from Equation 5. The agreement of estimated, K, and experimental, K_{exp}, values is sufficiently good for measuring higher shear rates with accuracy about 20%. The determined diffusivities, D, of ferricyanide ions were by 7 to 8% lower than the tabulated data on D_∞ [6].

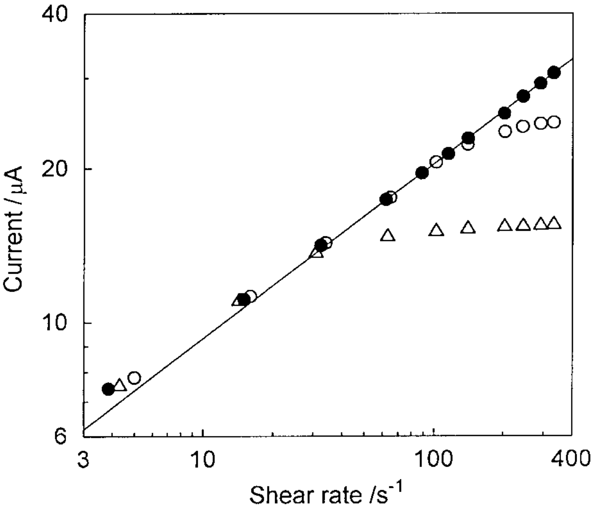

Fig. 3. I/γ calibration curve for the portable probe (dia. 0.5 mm) including the effect of an additional resistance, R: (△) 50, (○) 30 and (●) 0 kΩ.

3.3. *Measuring shear rates on high-speed rotating bodies*

The electrodiffusion technique [13, 14] was used for shear rate measurements on the rotating disc and pump impeller with diameters 176 and 170 mm, respectively. Three circular probes (dia. 0.5 mm) were embedded in the disc and twelve probes in the blades and disc of the impeller shown in Fig. 5. Obviously, it is impossible to calibrate the probes in a viscometric flow. The Cottrell coefficients, a_{exp}, were used to obtain estimates of K. An example of the transient

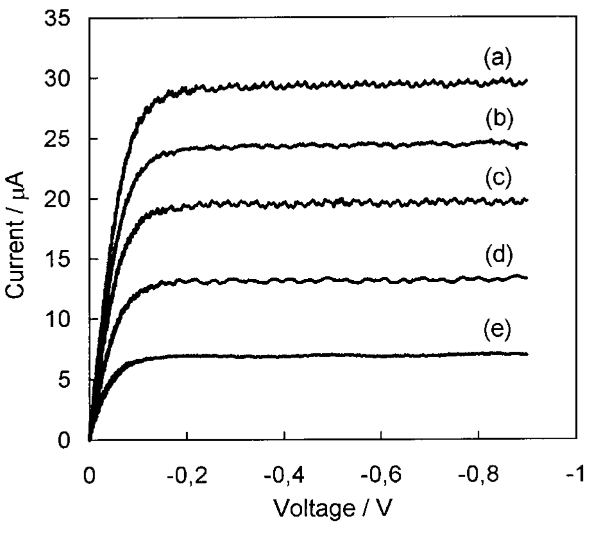

Fig. 2. Polarization curves for the portable probe (dia. 0.5 mm) at several shear rates, γ: (a) 330, (b) 200, (c) 100, (d) 30 and (e) $3\,\mathrm{s^{-1}}$.

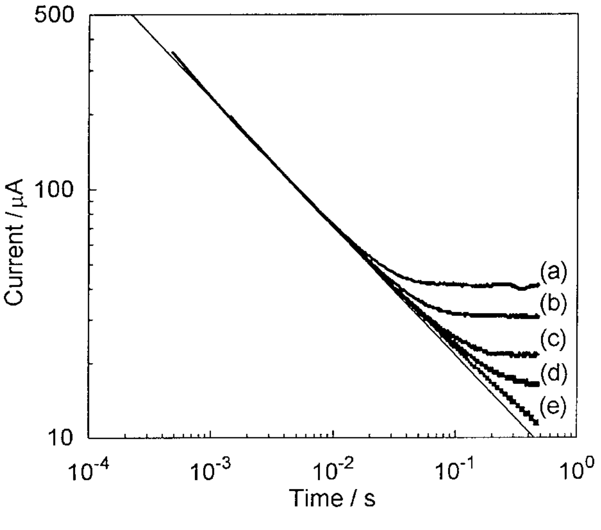

Fig. 4. Potentiostatic transient curves for the portable probe (dia. 0.5 mm) at several shear rates, γ: (a) 470, (b) 370, (c) 130, (d) 65 and (e) $0\,\mathrm{s^{-1}}$.

Table 1. Results of complete and partial calibrations

Probe	T /°C	a_{exp} /μA s$^{1/2}$	$10^{10}D$ /m^2 s^{-1}	$10^{10}D_\infty$ /m^2 s^{-1}	K /μA s$^{1/3}$	K_{exp} /μA s$^{1/3}$
Diam. 0.5 mm, portable	21	7.34	7.5	8.05	4.26	4.25
0.11 mm × 0.99 mm, portable	23	3.90	7.9	8.50	3.47	3.40
Diam. 0.5 mm, embedded in disc	20	7.14	7.2	7.83	4.15	–

experiment is given in Fig. 6 for the electrode embedded at a radius $d/2 = 40$ mm in the disc rotating with a speed of 65 rpm ($N = 1.08\,\mathrm{s}^{-1}$) in a baffled vessel. The value of K in Table 1 was estimated from a_{exp} in the same way as for the portable probes. For the estimated value $K = 4.15\,\mu\mathrm{A\ s}^{1/3}$ and experimental steady-state current $I_{\mathrm{L}} = 33.4\,\mu\mathrm{A}$ (see Fig. 6), it follows that $\gamma = 536\,\mathrm{s}^{-1}$ from Equation 1(a). This value is only by 5% lower than the local prediction for a high-speed rotating disc [18], $\gamma = 6.302\,(N^3d^2\rho/\eta)^{1/2} = 567\,\mathrm{s}^{-1}$. The scatter of the a_{exp} values for three electrodes in the disc was $\pm 0.2\,\mu\mathrm{A\ s}^{1/2}$. In the case of the impeller (Fig. 5), it was difficult to position the electrodes in the 4 mm thick impeller blade perpendicularly to the blade surface. Due to this difficulty, some electrodes had a larger area and a_{exp} had values $7.6 \pm 0.5\,\mu\mathrm{A\ s}^{1/2}$ at 20 °C.

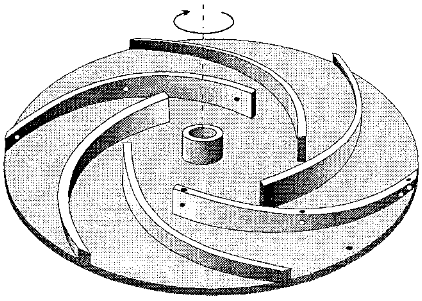

Fig. 5. Impeller with 12 ED friction probes (the 12th one is hidden on the opposite side).

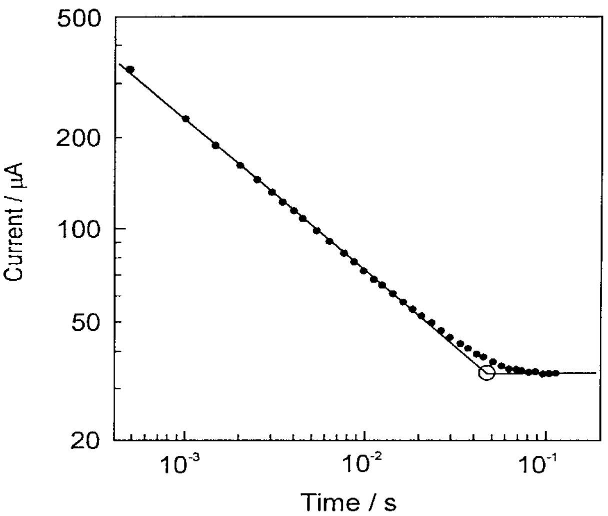

Fig. 6. Complete transient curve for the probe (dia. 0.5 mm) embedded in the rotating disc. The resulting parameters of the both asymptotes, $a_{\mathrm{exp}} = 7.14 \pm 0.2\,\mu\mathrm{A\,s}^{1/2}$, $I_{\mathrm{L}} = 33.4 \pm 0.8\,\mu\mathrm{A}$, are compared with other characteristics in Table 1.

3.4. *Additional ohmic resistance*

The ohmic losses cannot be completely eliminated, as they are concentrated in a region close to the working microelectrode. As predicted by recent theory [19], their presence is manifested by an overshoot of the transient currents above the Cottrell asymptote. This typical feature of the voltage-step transient for electrodes with active surface, unaffected by catalytic poisons, is shown in Fig. 7 for several values of the purposefully added outer resistance. The difference between predicted and observed courses is probably due to the neglected faradaic resistance which is analysed in [20]. It follows from the experiment with no additional resistance that the inner resistance of the ED probe is a small fraction of 1 kΩ. The corresponding steady-state behaviour is shown in Fig. 3.

3.5. *Poisoned electrodes*

The polarization curves of electrodes deactivated by a poisoning are well-known [3]. Two other working characteristics of a strongly poisoned circular portable ED probe (dia. 0.5 mm) are shown in Figs 8 and 9. Even the poor characteristics shown can be treated in a quantitative way, when using a proper transport model [20, 21].

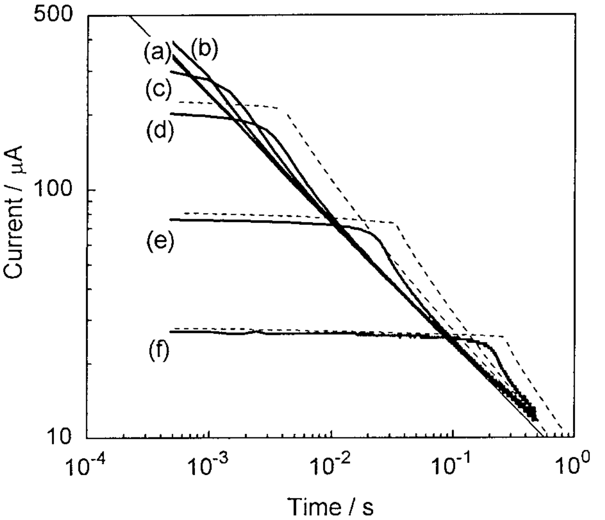

Fig. 7. Early stage of the transient process at $\gamma = 0\,\mathrm{s}^{-1}$ with additional resistance R in series with the ED cell: (a) 0, (b) 1, (c) 2, (d) 3, (e) 10 and (f) 30 kΩ. The dashed lines show the theoretical asymptotes [19] accounting for the ohmic losses.

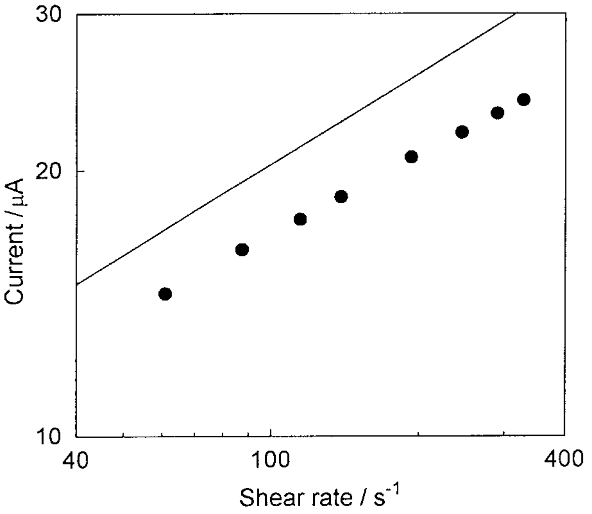

Fig. 8. Effect of poisoning on the I/γ calibration curves. Straight line, taken from Fig. 3, shows the behaviour of the nearly perfect ED probe. Solid points: data for the poisoned ED probe of the same size.

3.6. *Dynamics of an ED probe under fluctuating flow*

The frequency response of ED friction probes on hydrodynamic fluctuation [5, 11, 22–24] can easily be predicted from the knowledge of potentiostatic transient time t_0. It is evident from Fig. 10, that the formula

$$I^*/\gamma^* \equiv H^* \approx (3 + \mathrm{i}\omega t_0)^{-1} \qquad (7)$$

approximates the exact impedance both for the rectangular and circular ED friction probes. Differences between the impedance characteristics for rectangular and circular probes become indistinguishable when using the generalized variable ωt_0. Nearly the same results for the impedance were obtained in the case of regular wavy film flow, where the wall shear rate fluctuations were caused by surface waves [11, 26].

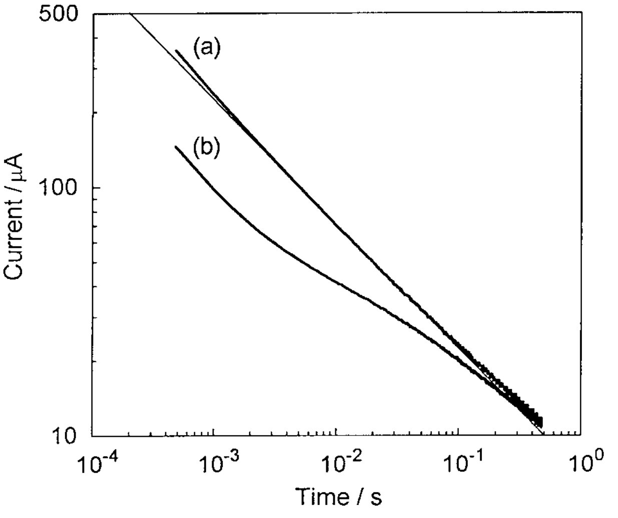

Fig. 9. Effect of poisoning on the voltage-step transient curves at $\gamma = 0\,\mathrm{s}^{-1}$. (a) Data for the nearly perfect ED probe (b) data for the poisoned ED probe of the same size. Thin solid line shows the corresponding Cottrell asymptote.

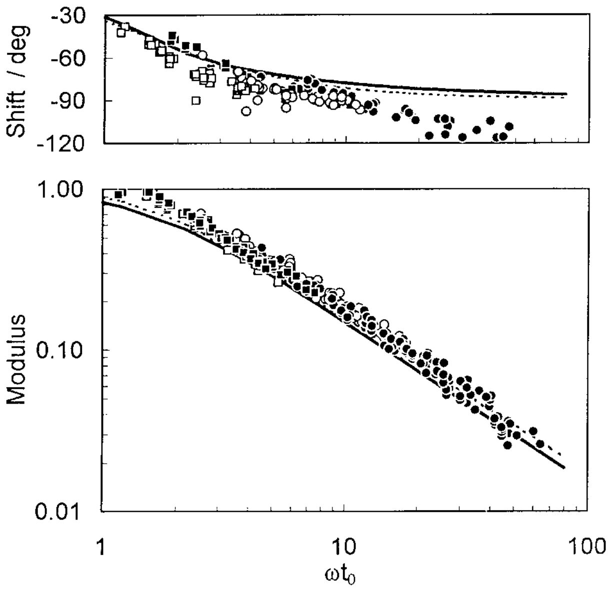

Fig. 10. Experimental data on reduced ED impedances, $H^*(\omega)/H^*(0)$, of rectangular (□, ■) and circular (○, ●) ED probes for CMC (□, ○) and glycerol (■, ●) solutions in a nonwavy film flow down the oscillating plate [24]. Dashed lines: exact theory [23]. Solid lines: approximate theory according to Equation 7.

4. Conclusions

The transient voltage-step experiment is a convenient method for checking the readiness of ED friction probes for hydrodynamic measurements, especially for higher shear rates.

The calibration coefficient, K, in the Leveque formula, $I = K\gamma^{1/3}$, determined by calibration experiments at low shear rates, can be safely estimated, corrected for changes in experimental conditions, and extrapolated, using the Cottrell asymptote from the partial calibration of ED friction probes.

The small (dia. 0.5 mm) circular electrodes can be safely used for hydrodynamic measurements for shear rates as high as $10^6\,\mathrm{s}^{-1}$. The electrodes are sufficiently smooth for these measurements when polished with emery paper (grit size 15 μm).

Ohmic losses and poisoning of the electrode surface manifest themselves clearly by the unusual transient and steady characteristics.

Two asymptotic parameters, I_L and a, of the complete transient calibration experiment provide sufficient information (the potentiostatic transient time t_0) for the correction of the fluctuating ED signal on the dynamics of ED friction probes.

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