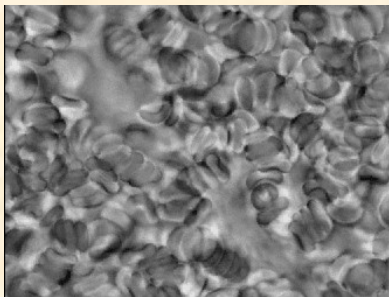
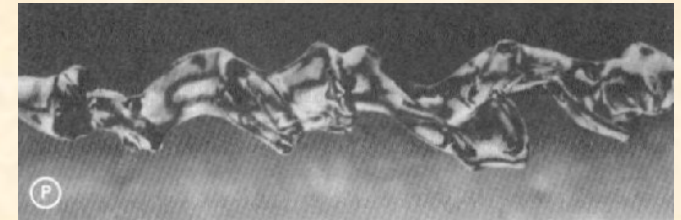
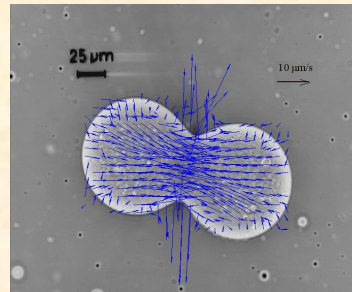
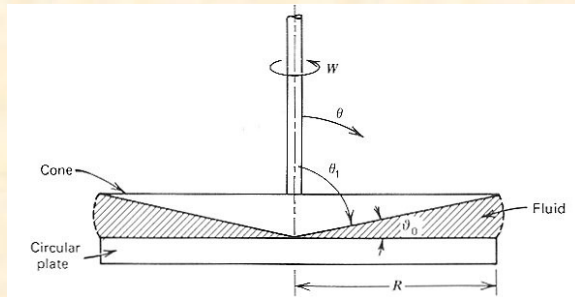
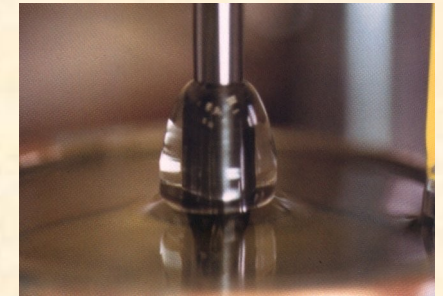


# Introduction à la rhéologie



**C. VERDIER**

Directeur de Recherches C.N.R.S.

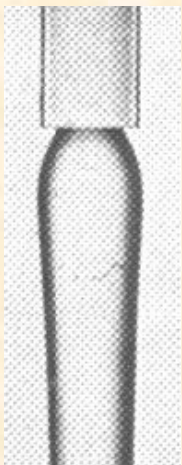
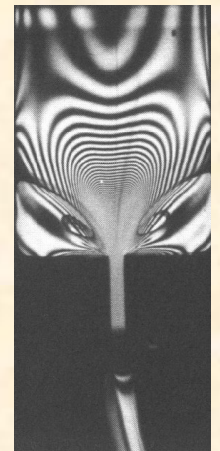


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**Document de cours :**

<http://www-liphy.ujf-grenoble.fr/pagesperso/verdier/>

>Teaching>Notes; Article; Présentation + exams



# Plan

**I. Définitions et contexte scientifique**

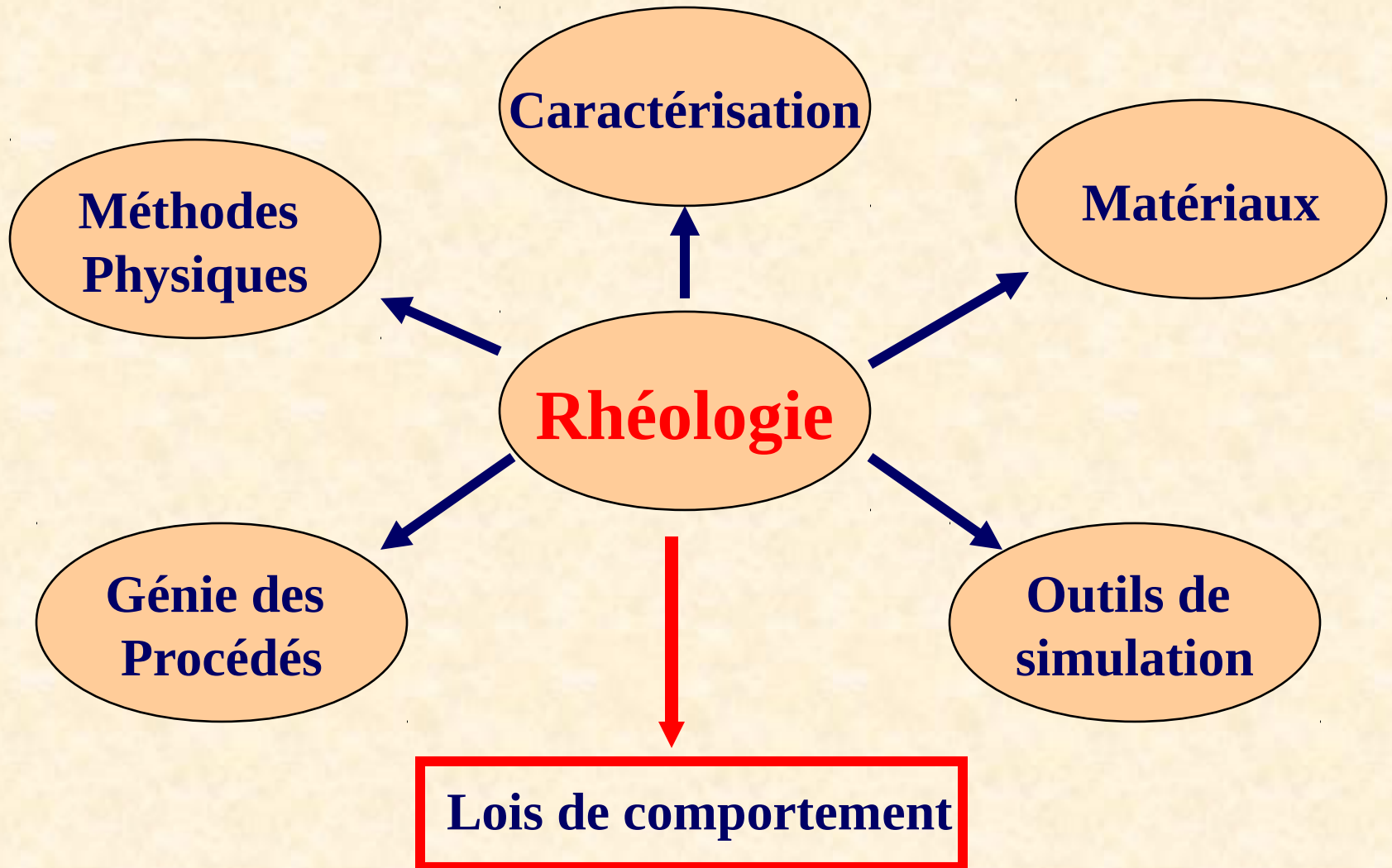
**II. Phénomènes particuliers**

**III. Mesures expérimentales**

**IV. Lois de comportement**

# I. Définitions et contexte scientifique

- **Rhéologie** = rheos (écoulement)  
+ logie (étude de)
- Science de l'écoulement de la matière des **contraintes** qu'il faut lui appliquer pour créer un **écoulement** (déformation) particulier(e)
- Domaine **pluridisciplinaire**
- **Applications** : Matériaux divers, procédés

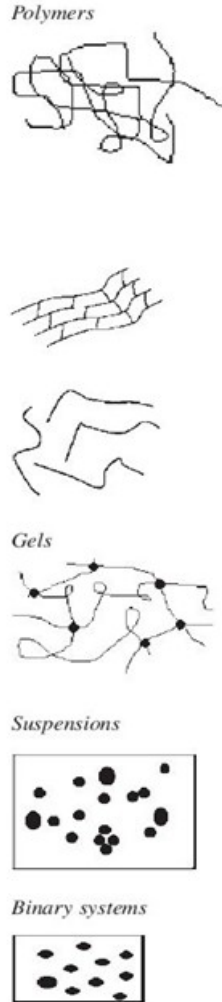


# Pluridisciplinarité de la rhéologie

- **Techniques expérimentales** : viscosité, élasticité, visco-élasticité, visco-plasticité
- **Rhéométrie** : cisaillement, élongation
- Propriétés de surface et de volume
- **Méthodes numériques** particulières
- Lois de comportement et **modélisation**
- Apport de la **physique** : microstructure

# Matériaux étudiés

- **Polymères** et dérivés (mélanges de polymères, alliages, polymères chargés, etc.)
- **Suspensions** : microniques, colloïdales, diphasique, cristaux liquides, émulsions
- **Gels** physiques, chimiques, biologiques
- Matériaux **viscoélastiques/viscoplastiques**
- **Divers** : pâtes, huiles, graisses, produits agroalimentaires, matériaux biologiques



# Procédés

- **Ecoulements** (hydraulique, méc. des fluides)
- **Fabrication** : extrusion, injection, moulage
- **Mise en forme, mélange** (2 composants ou plus)
- **Instabilités** : surface libre, jets
- **Divers** : enduction (peinture), couchage, adhésion (pelage, tack), etc.
- **Microfluidique, microrhéologie**

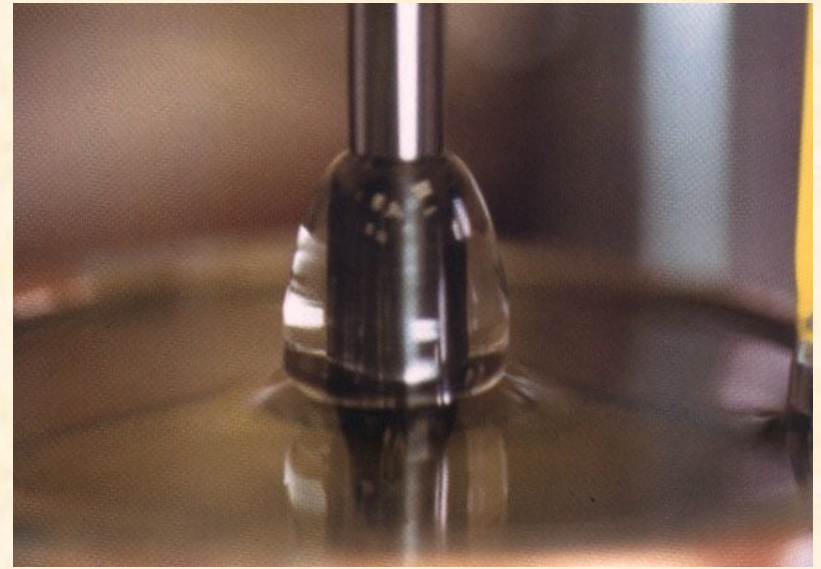
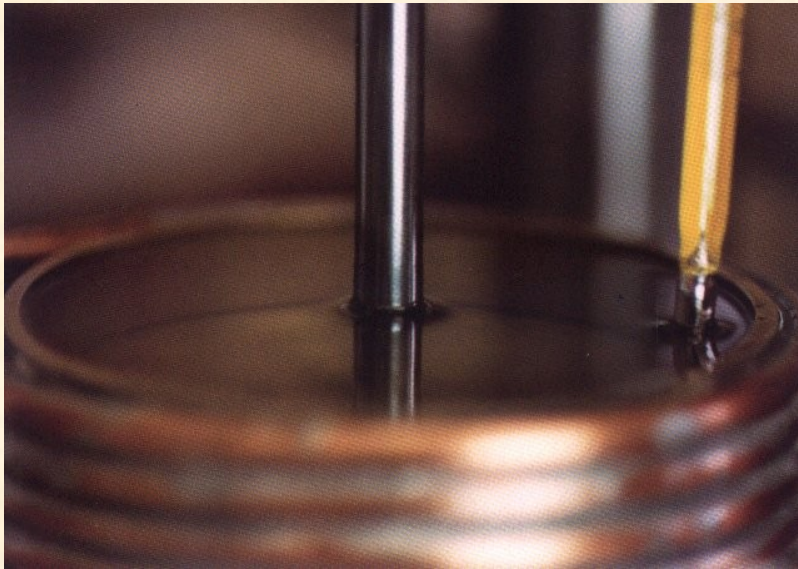
# Secteurs industriels concernés

- **Chimie** (polymères, synthèse)
- **Industrie pétrolière** (extraction)
- **Agro-alimentaire** (pâtes alimentaires)
- **Automobile** (graisses), **BTP** (ciment, béton)
- **Biotechnologies** (systèmes biologiques)
- **Cosmétiques** (suspensions)



## II. Phénomènes particuliers

### Effet Weissenberg



Joseph, 1977

Montée de liquide non-newtonien sur une tige  
en rotation

# Courbes de viscosité

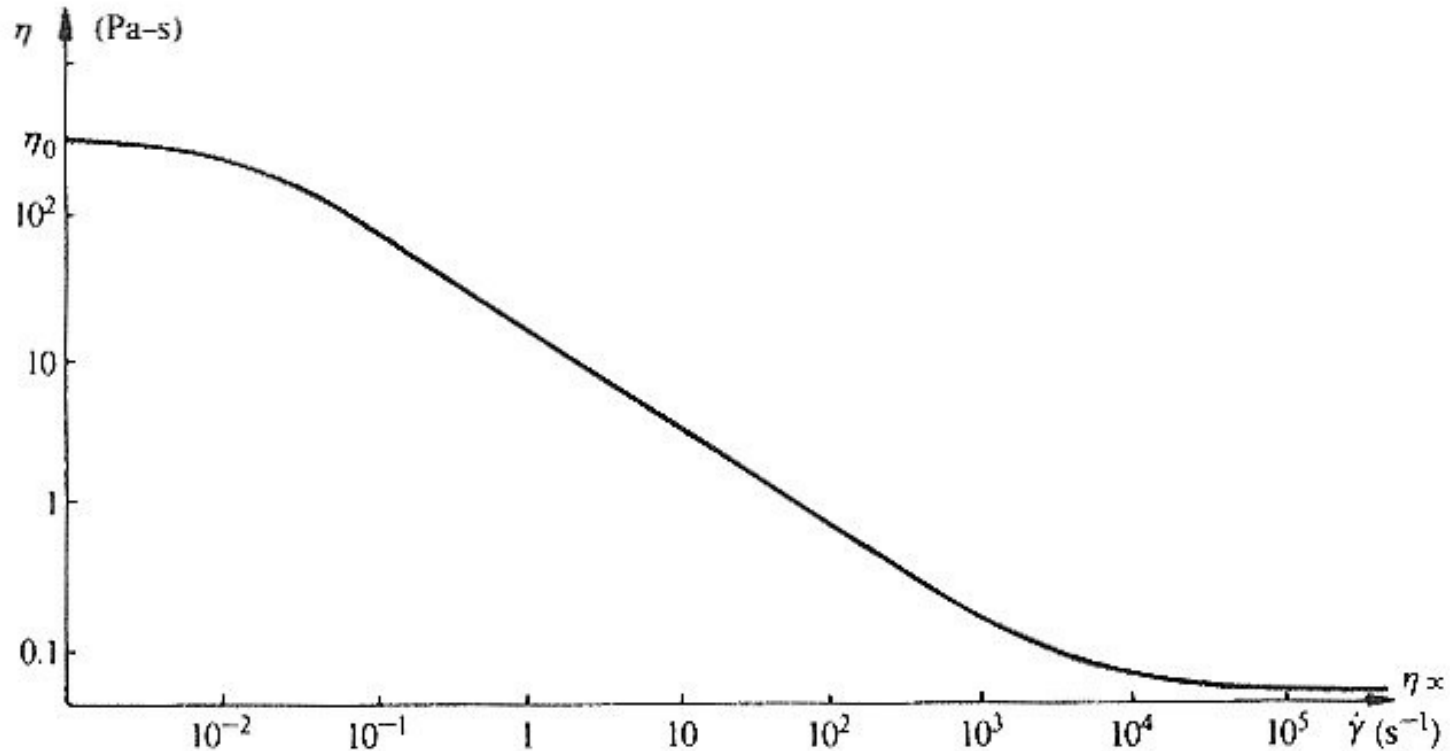
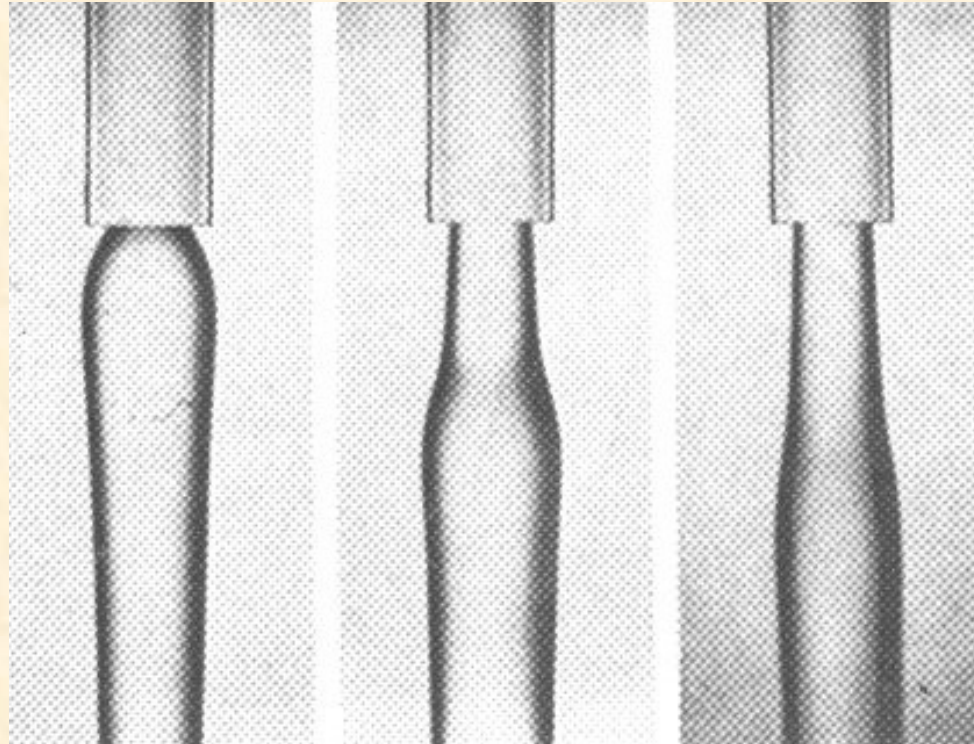


Fig. 1.2 Shear-thinning in a typical non-Newtonian fluid, Separan AP-30 in glycerol water.

**Dépendance en fonction du gradient de vitesse  
(solution de polymère)**

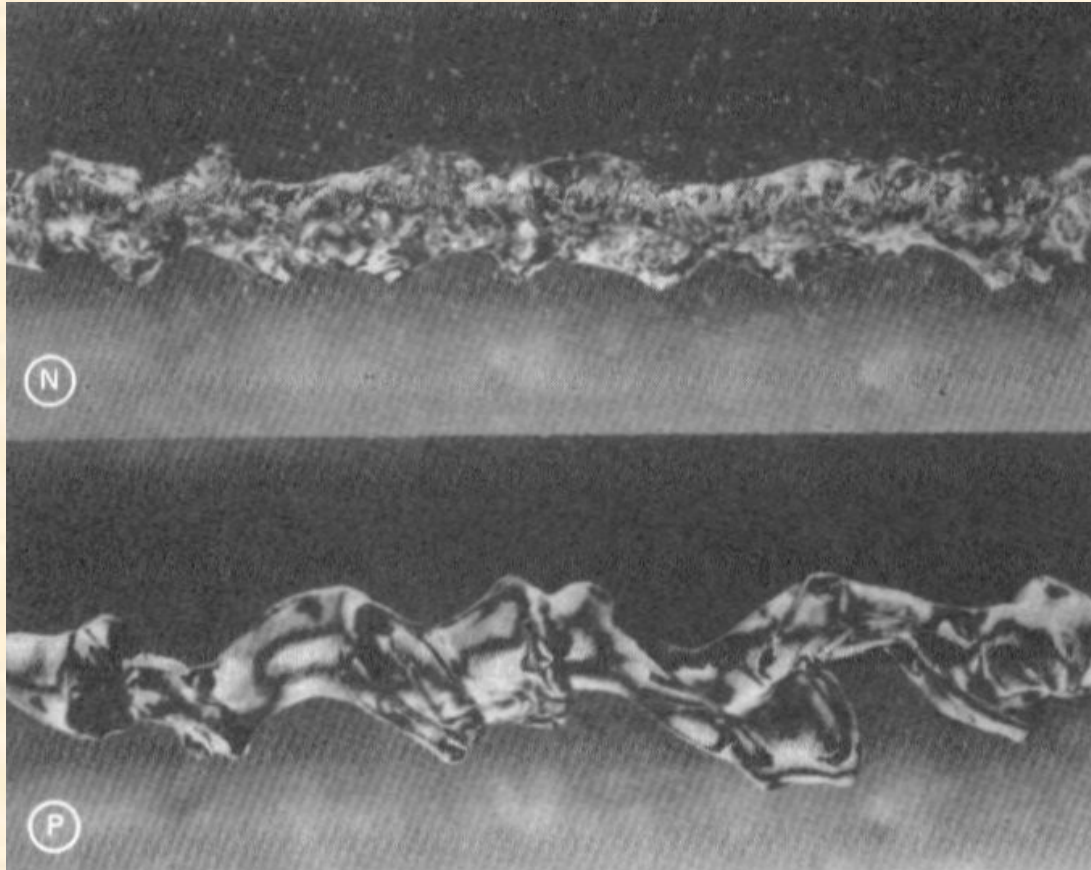
# Gonflement en sortie de filière



Giesekus, 1968

Gonflement en sortie de filière et  
gonflement différé

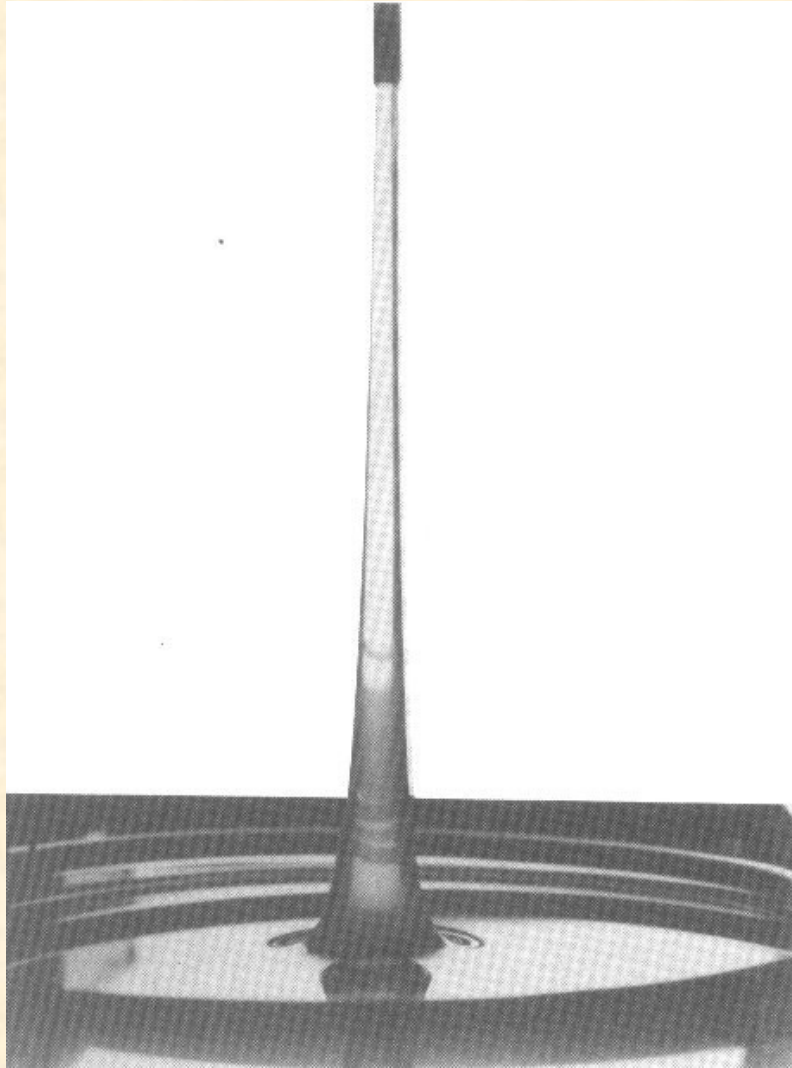
# Jets newtoniens/non-newtoniens



Hoyt & Taylor, 1977

Rupture d'un jet N=Newt., P= Non Newt.

# Siphon sans tube



Siphon : polymère de  
Poids moléculaire élevé

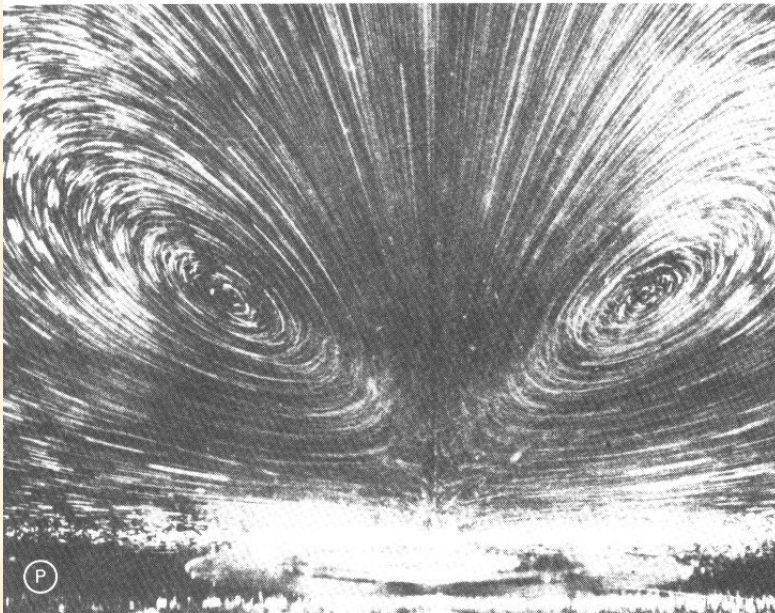
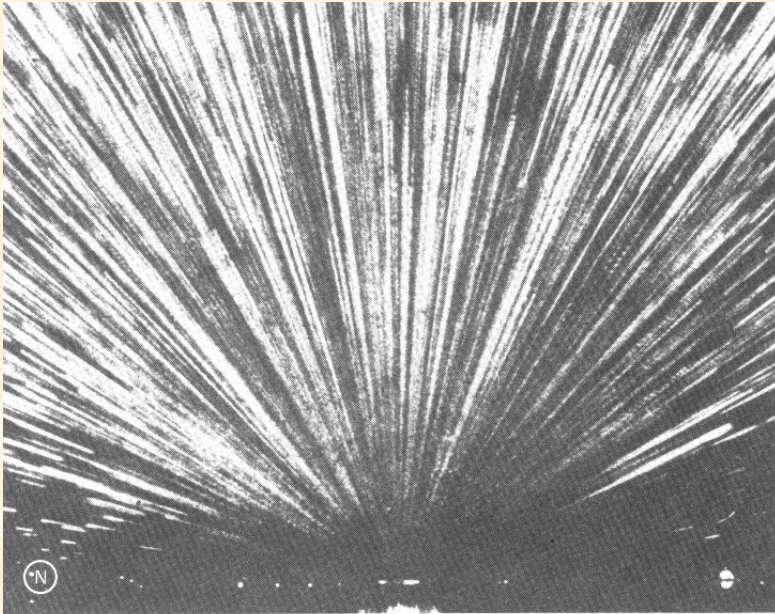
Peng et al., 1976

Application au  
pompage du pétrole

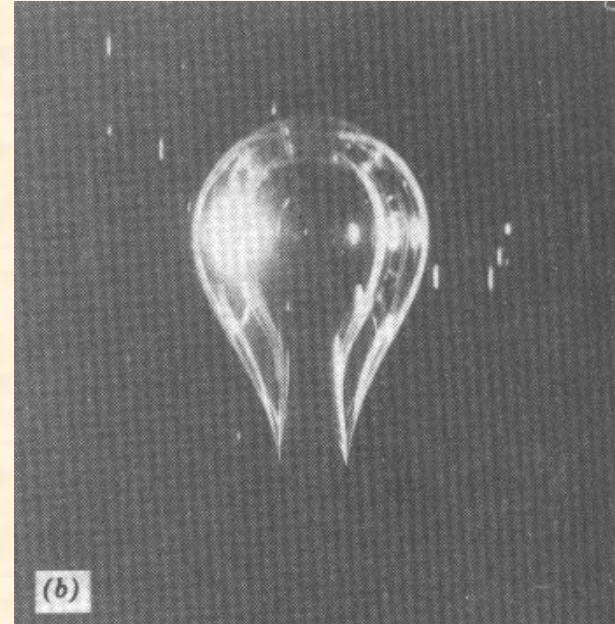
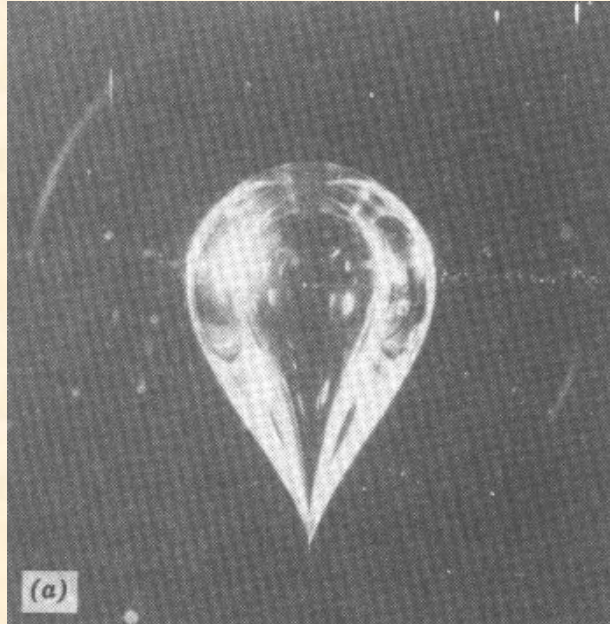
# Écoulement dans une contraction

Metzner et al., 1969

- Présence de recirculations
- Instabilités



# Bulles dans les fluides complexes



**Bulles en ascension dans une solution de polymère vue de deux cotés**

# Explication des phénomènes

- Effet des **contraintes normales** (même en cisaillement)
- **Longueur** des chaînes : cohésion des polymères
- **Echelle de temps** importante :

Nombre de Deborah  $De = \lambda/t_{\text{exp}}$

( $\lambda$  = temps caractéristique,  $t_{\text{exp}}$  temps de l'expérience)

- **Lois de comportement** particulières



# III. Mesures expérimentales

- Rhéométrie de **cisaillement**  $\sigma = \sigma_{12}(\dot{\gamma})$



$$U_z = U_{\max} (1 - (r/R)^2)$$

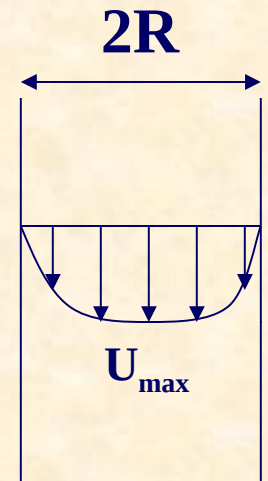
$$Q = \pi U_{\max} R^2/2$$

$R$  = rayon du tube

$$\dot{\gamma} = 4Q/\pi R^3$$

$$\Delta P = 8\eta LQ/\pi R^4 \implies \text{et } \eta(\dot{\gamma})$$

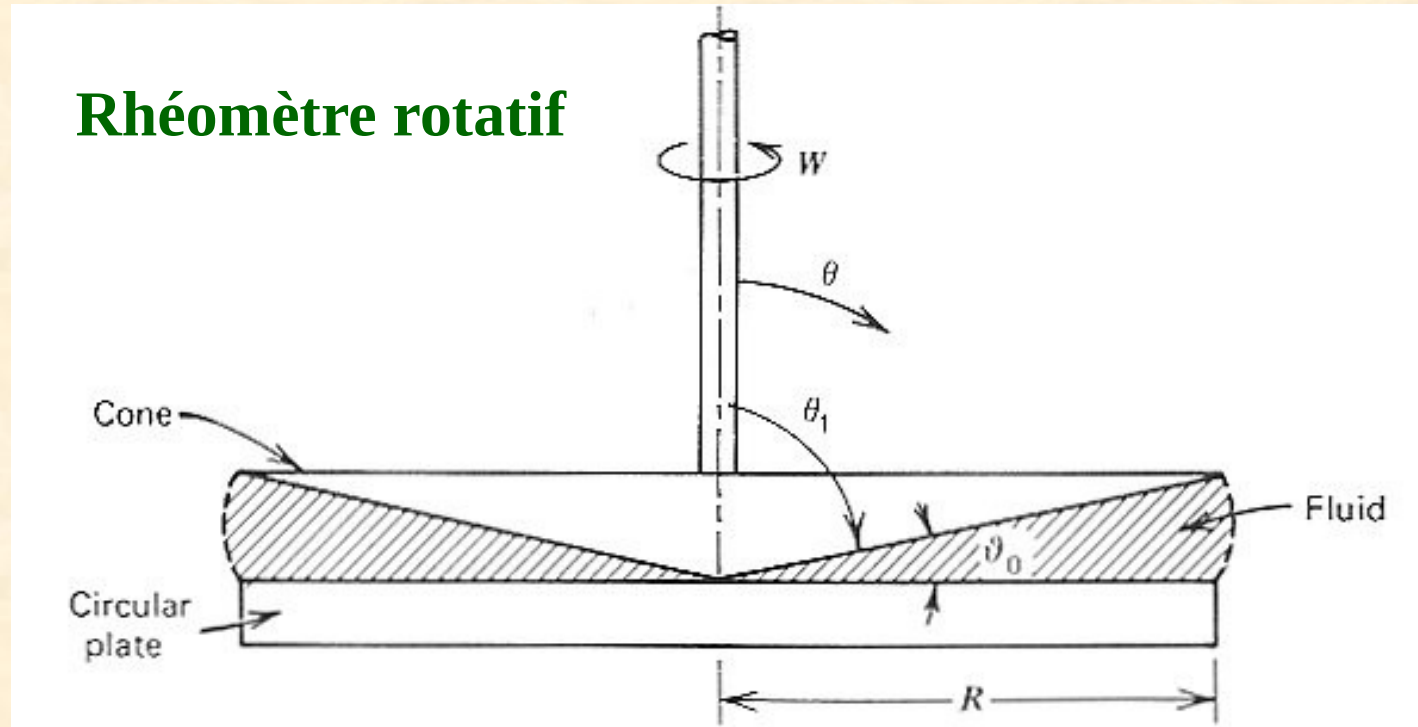
$$\text{Calcul : } -dp/dz = (d\sigma/dr + \sigma/r)$$



Tube de **Ubbelohde**  $t/t_0 = \eta/\eta_0$

**Rhéomètre capillaire**

- Rhéométrie de cisaillement  $\sigma = \sigma_{12}(\dot{\gamma})$



**Avantage : taux de cisaillement constant**  
**Inconvénient : mise en place échantillon**

# Résultats classiques en cisaillement

## A - Mesures dynamiques (déf. sinusoïdales)

=> Petites déformations  $\hat{\gamma} \ll 1$

$$\gamma = \gamma_0 \sin(\omega t)$$

### Module complexe

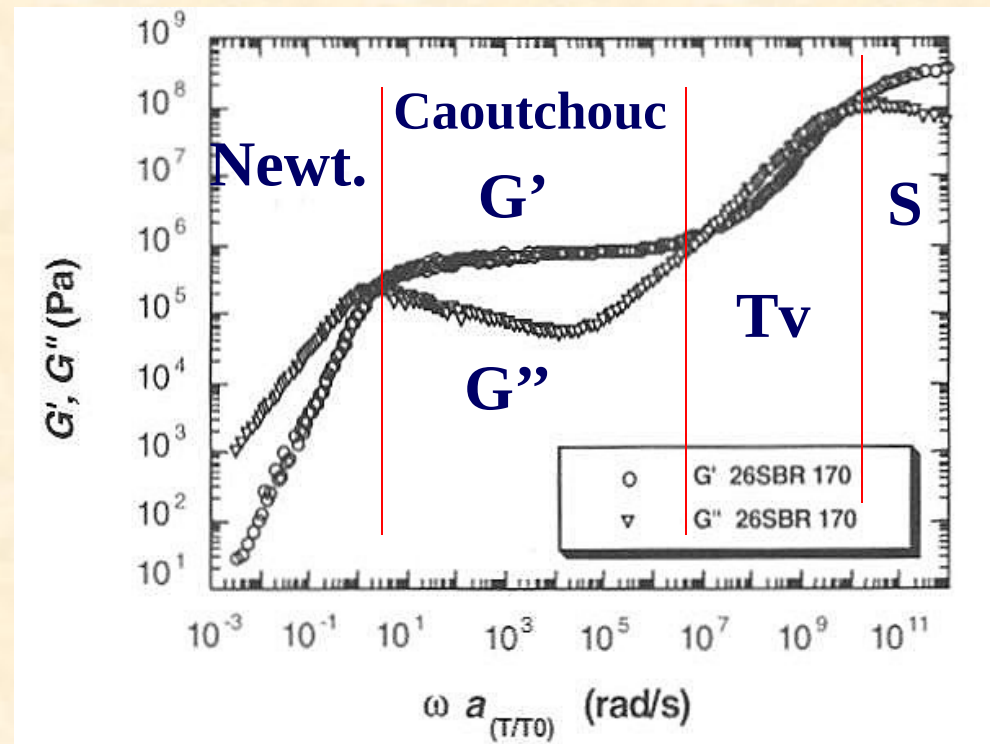
$$G^* = i\omega \int_0^\infty G(s) e^{-i\omega s} ds$$

### Viscosité complexe

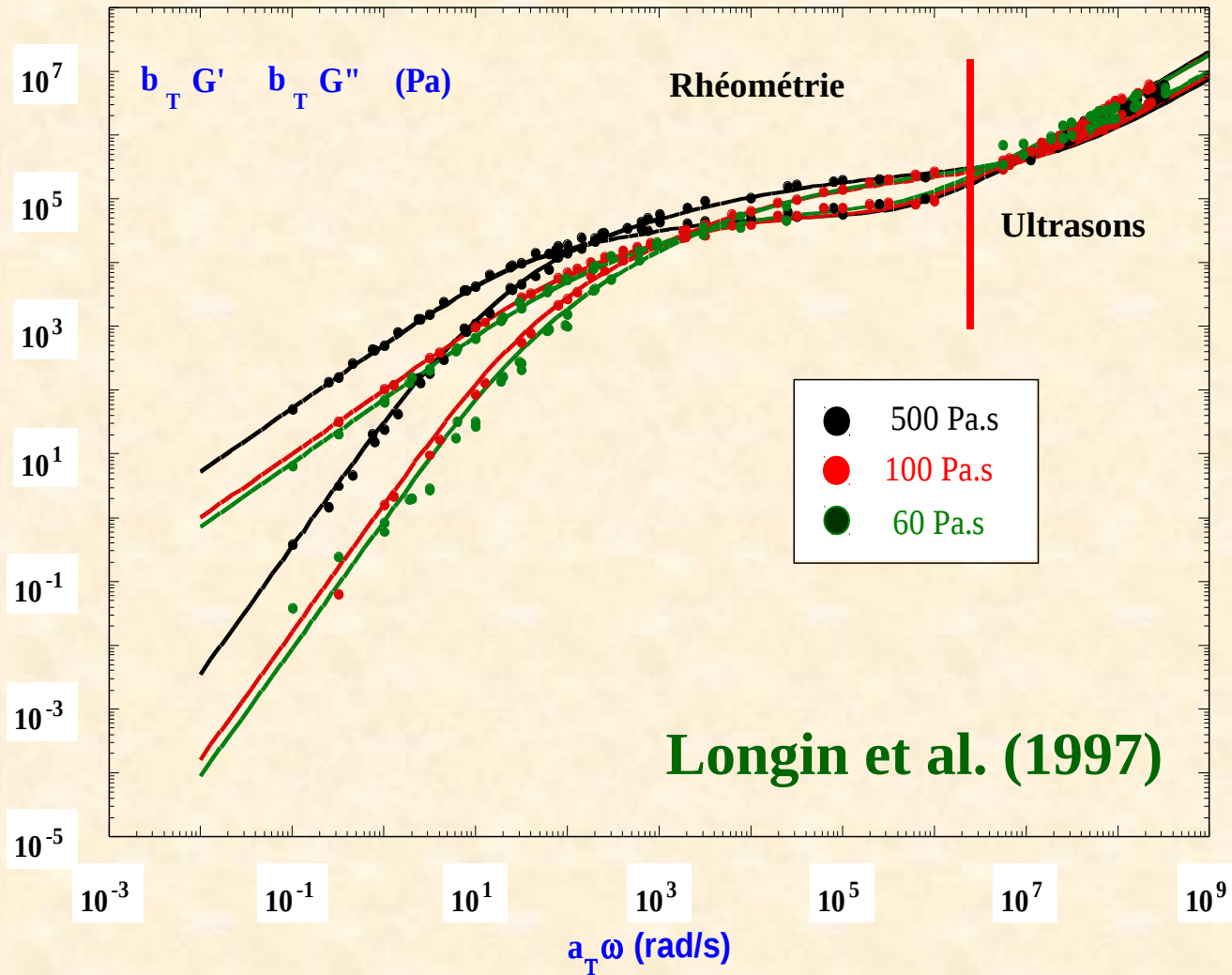
$$\eta^* = \eta' - i\eta'' = \frac{G''}{\omega} - i \frac{G'}{\omega}$$

Contrainte

$$\tau = \tau_0 \sin(\omega t + \varphi)$$



# Superposition temps-température : PDMS

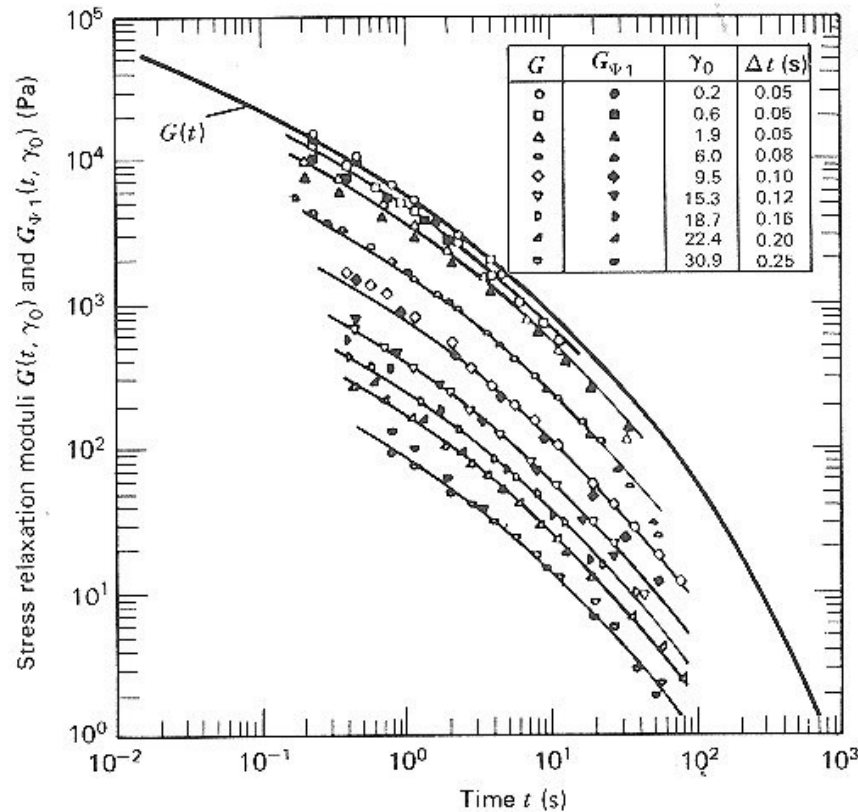


# B - Cisaillement transitoire (relaxation)

$$G^* = i\omega \int_0^{\infty} G(s) e^{-i\omega s} ds$$

$$G(t) = \sum_{i=1}^n G_i e^{-\frac{t}{\lambda_i}}$$

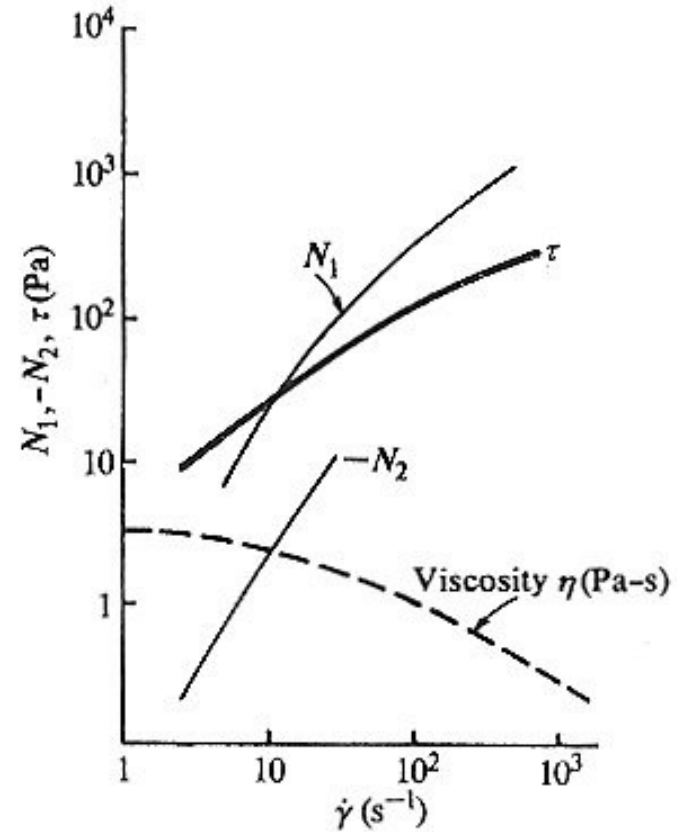
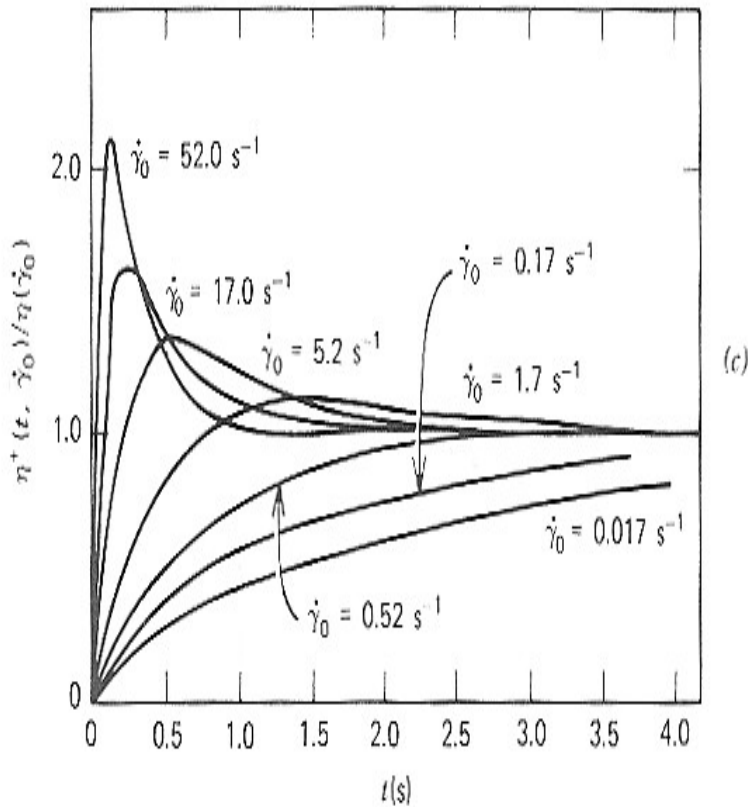
$G(t, \gamma_0)$  (Pa)



Relaxation

$t(s)$

## C- Courbes de viscosité $\eta = \eta(\dot{\gamma})$



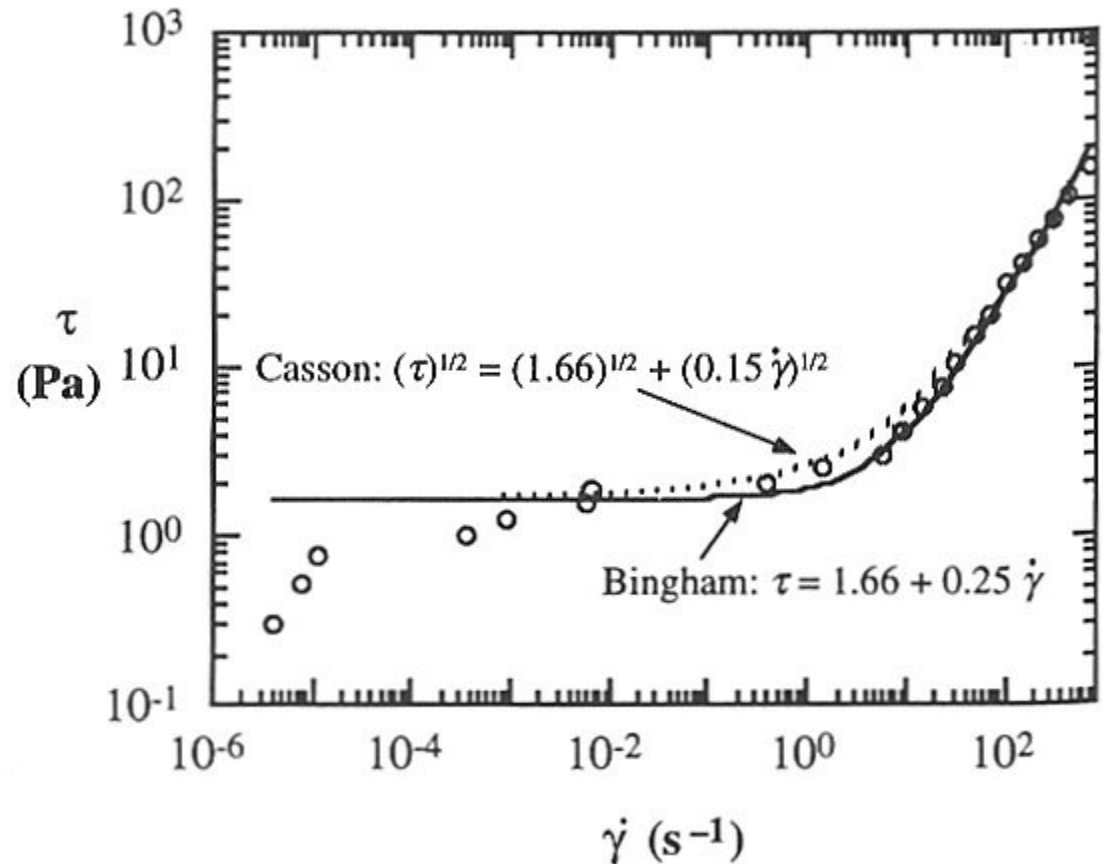
**Essai en transitoire**  
(montée en contrainte  
« dépassement »)

**Courbes en fonction du  
gradient de cisaillement**

# Fluides à seuil : suspensions, gels, etc.

Figure 2.5.4.

Comparison of Bingham and Casson fits to the iron oxide suspension data over the entire range of experimental data obtained; parameters for the Bingham model are  $\eta = 0.25 \text{ Pa}\cdot\text{s}$  and  $\tau_c = 1.66 \text{ Pa}$ , while for the Casson model they are  $0.15 \text{ Pa}\cdot\text{s}$  and  $1.66 \text{ Pa}$ , respectively.



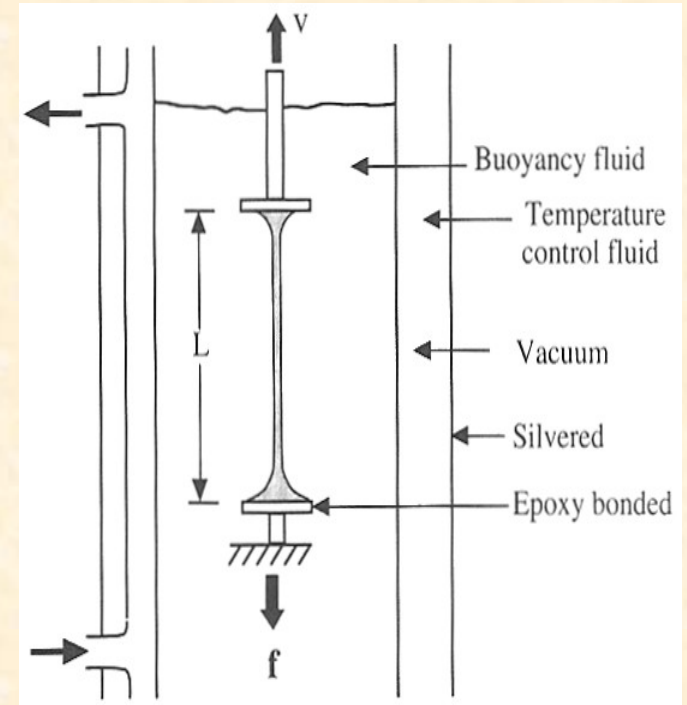
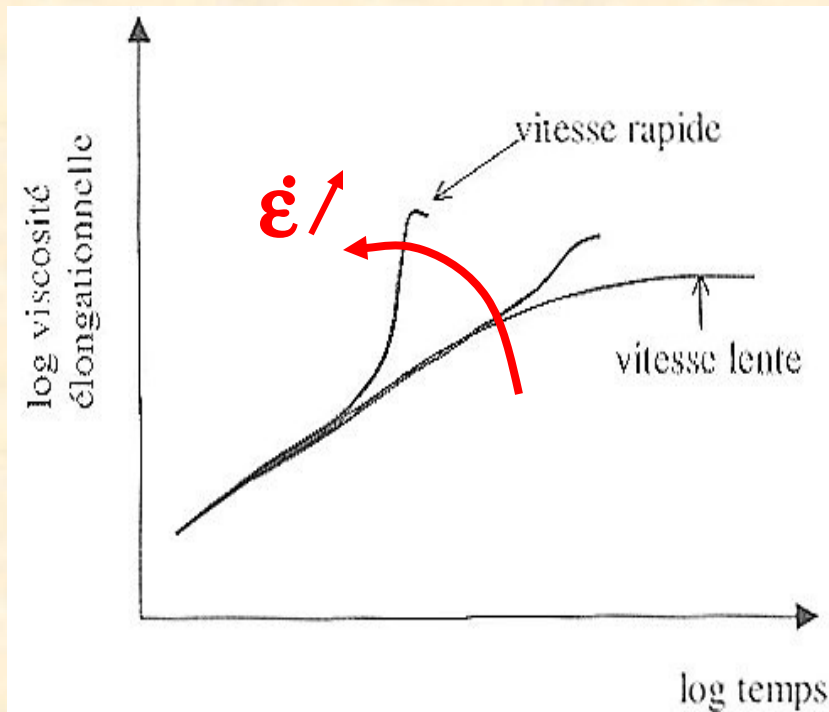
## Viscosité d'une suspension

$$\eta = \eta_s (1 + 2.5 \phi + k \phi^2 + \dots) \quad \text{Einstein, Batchelor (1972)}$$

$$[\eta] = \lim_{\phi \rightarrow 0} (\eta - \eta_s) / \phi \eta_s \quad \text{viscosité intrinsèque} = [\eta]$$

• Rhéométrie **élongationnelle**  $\sigma_{11} - \sigma_{22}(\dot{\epsilon})$

Taux d'élongation constant  $\dot{\epsilon}$   
 $\sigma_{11} - \sigma_{22}$  fonctions de  $t$  et  $\dot{\epsilon}$



**Viscosité élongationnelle**

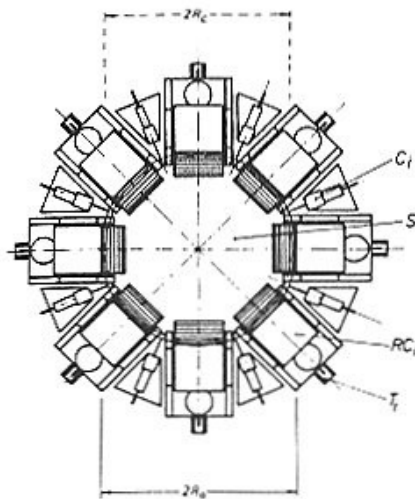
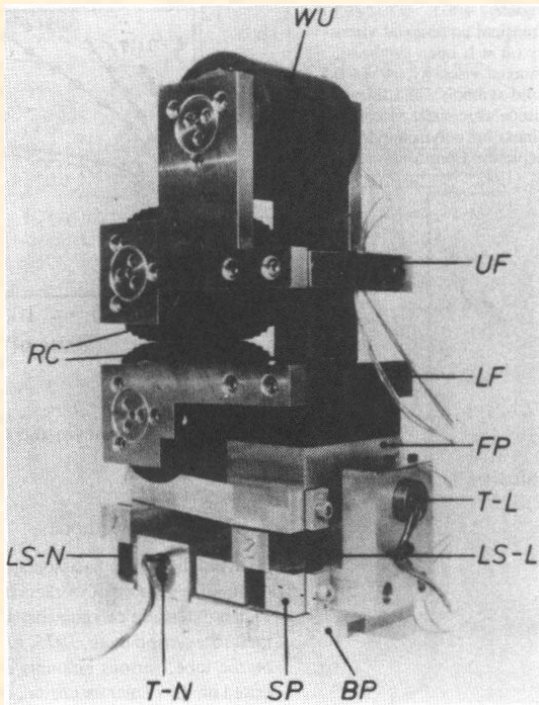
$$\eta_E^+(t) = (\sigma_{11} - \sigma_{22}) / \dot{\epsilon}$$



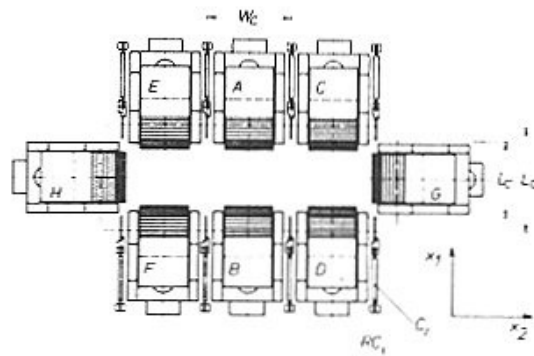
# Rhéométrie **élongationnelle**

- Elongation multiaxiale
- Machine à rouleaux
- Systèmes très visqueux

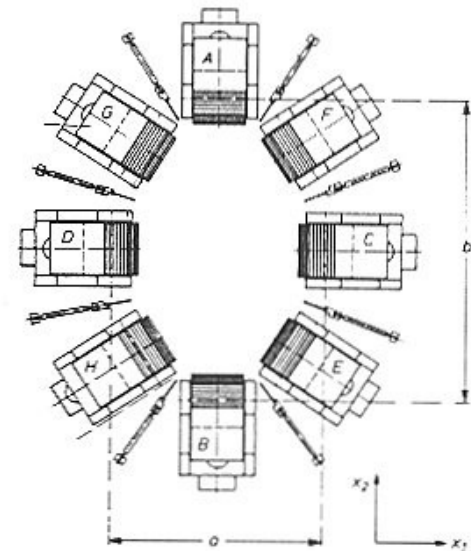
Meissner (1982)



(a)

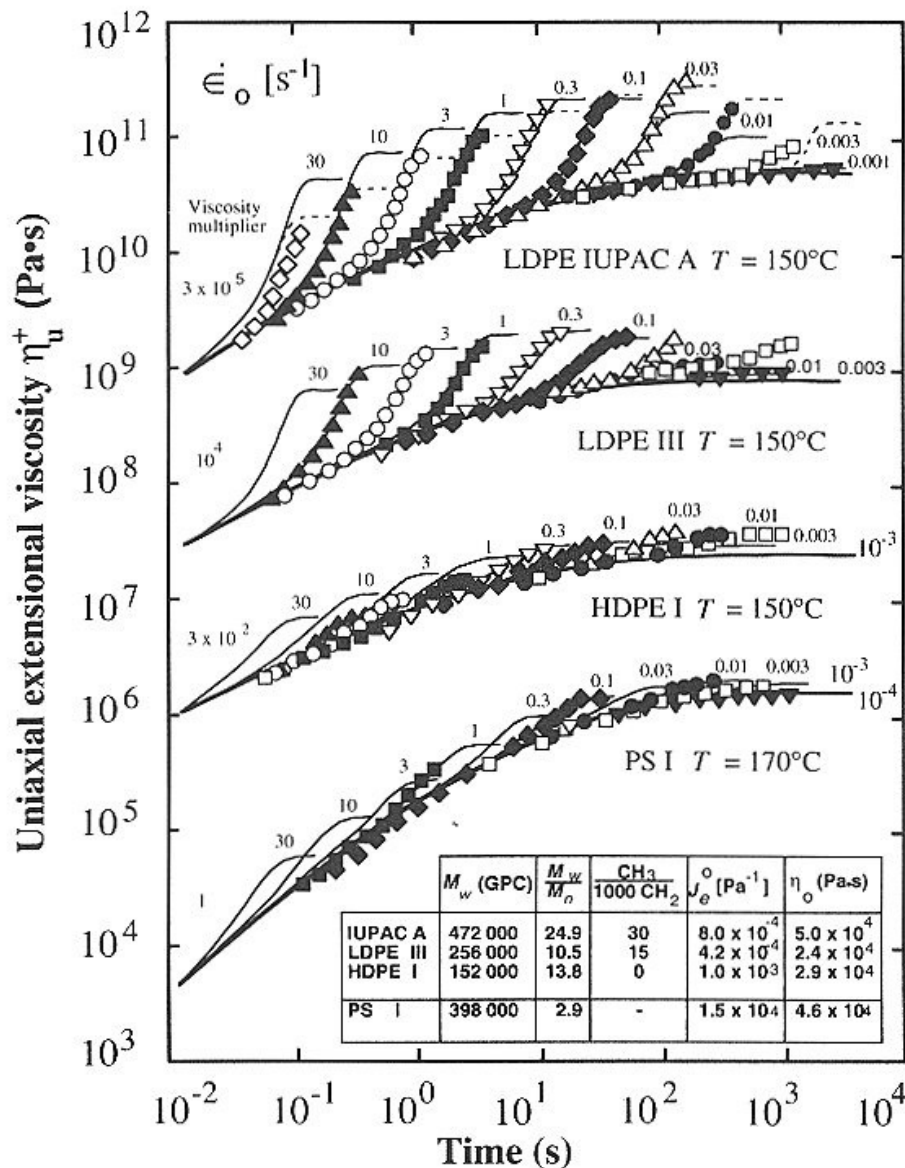


(b)



(c)

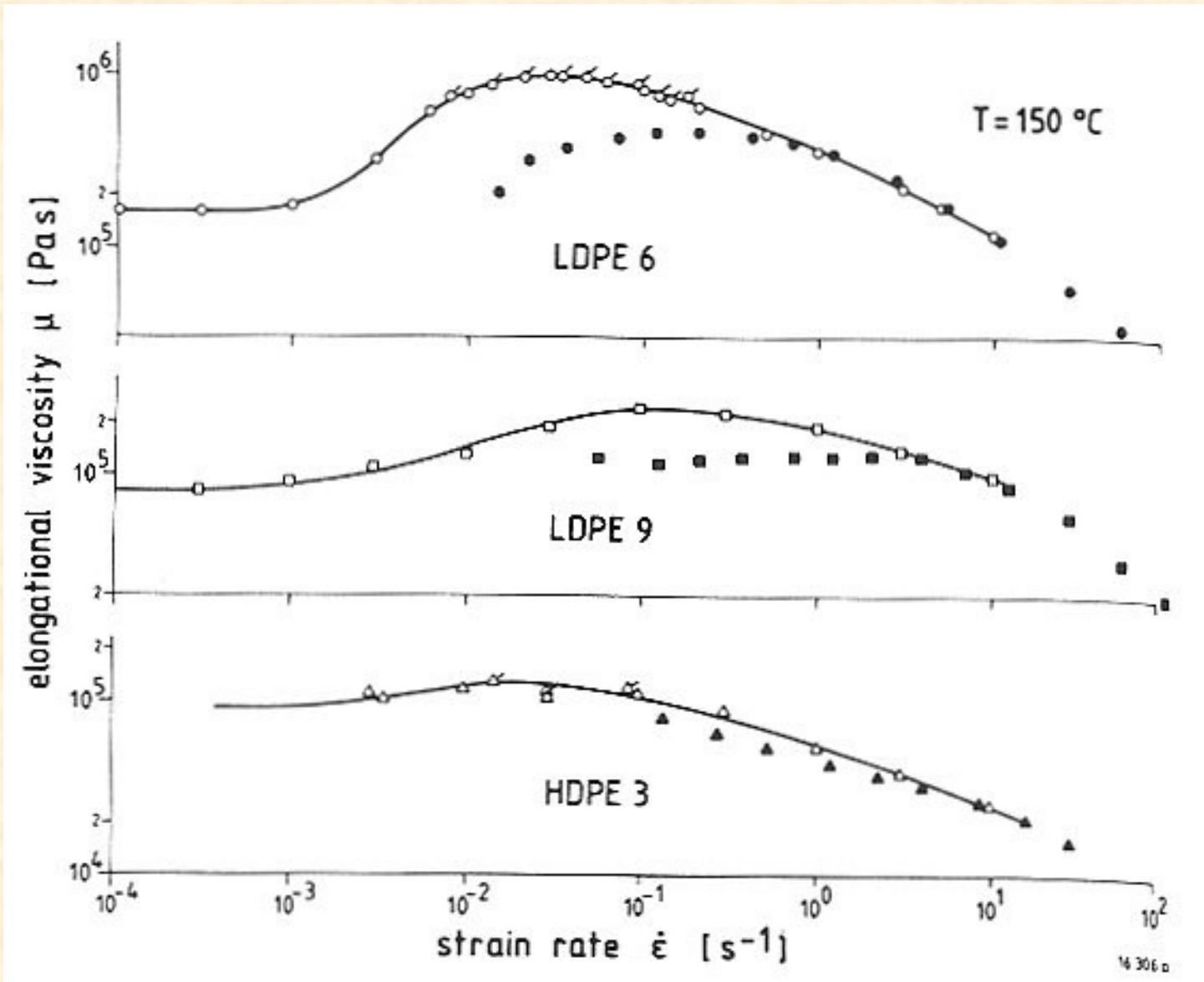
# A- Courbes de viscosité $\eta_E^+(t)$



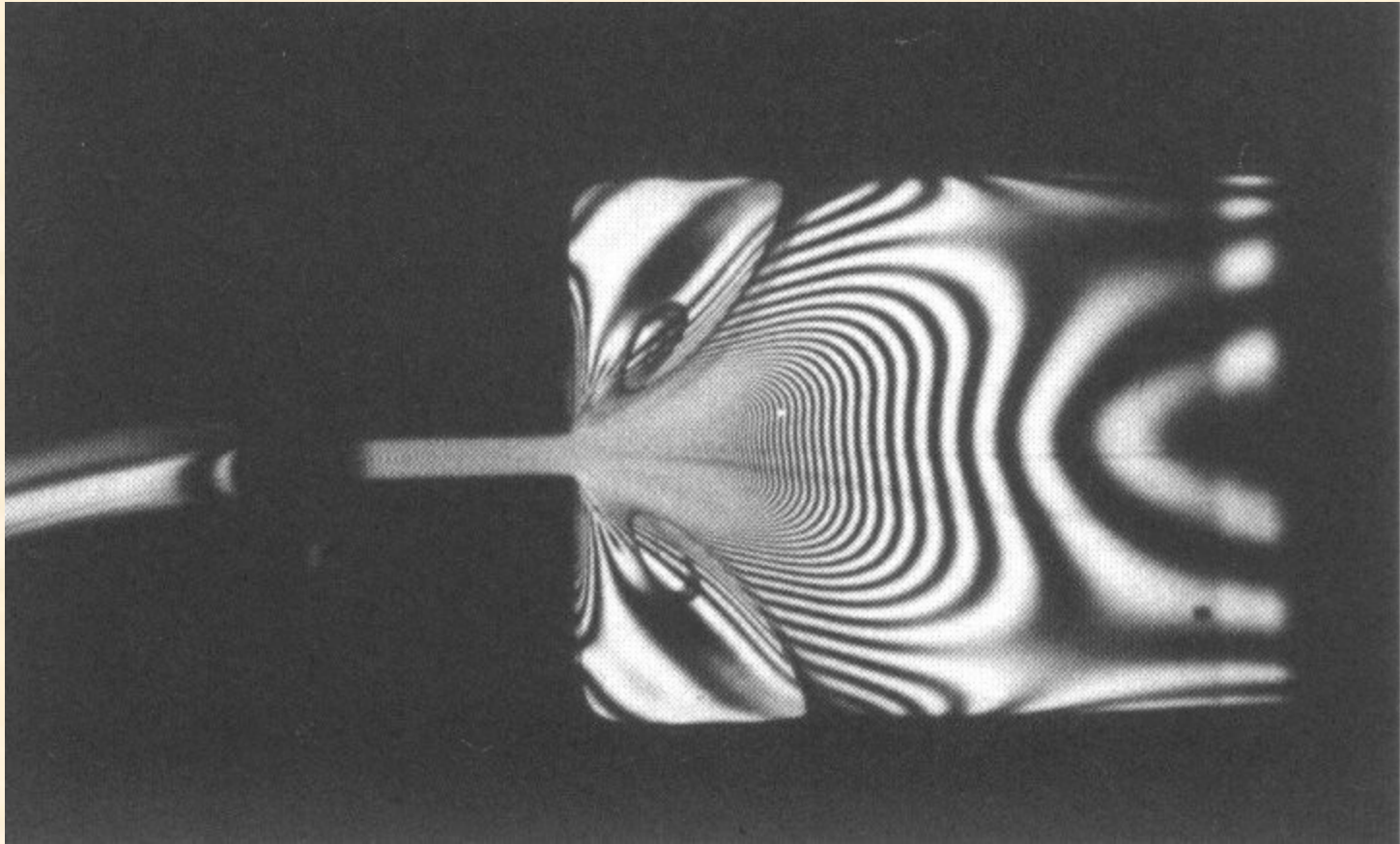
Laun (1984)

- **Durcissement** « strain hardening »
- **Régimes** permanent (petit  $\dot{\epsilon}$ ) ou divergence (grand  $\dot{\epsilon}$ )
- Indépendant du type de polymère
- Mesures **difficiles**

# B - Courbes de viscosité $\eta_E(\dot{\epsilon})$

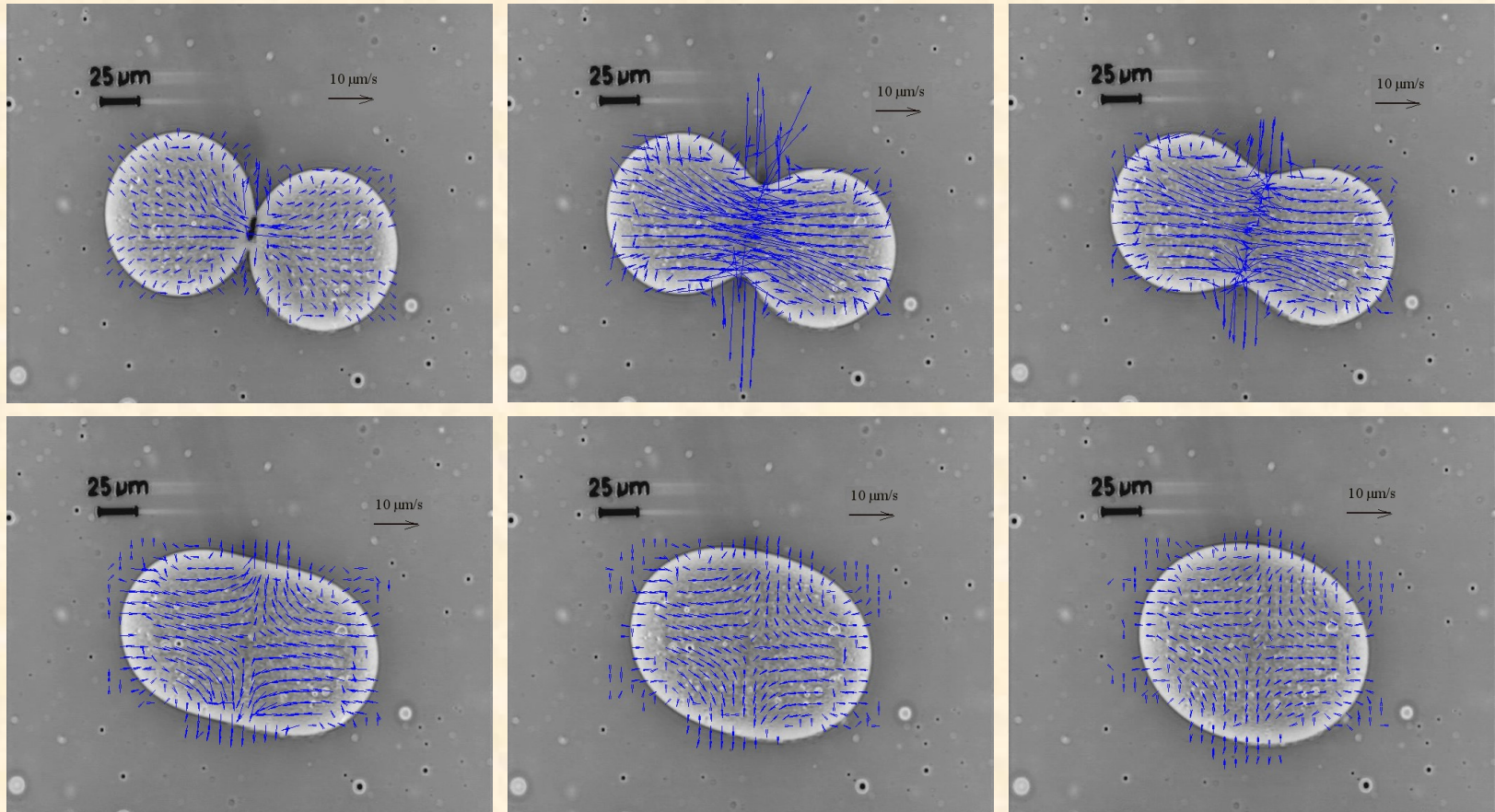


# Techniques particulières : biréfringence



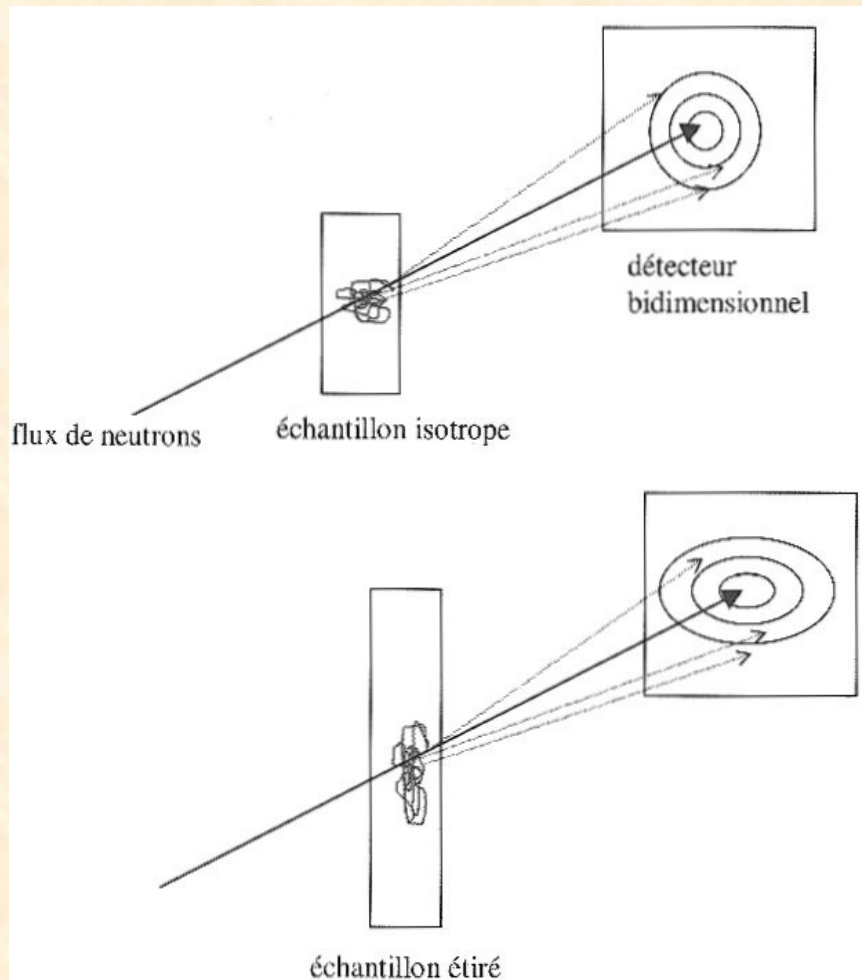
**Polystyrène dans une contraction :  
recirculations, et **isochromes**  $\sigma_{11} - \sigma_{22} = \text{constante}$**

# Techniques physiques particulières : PIV



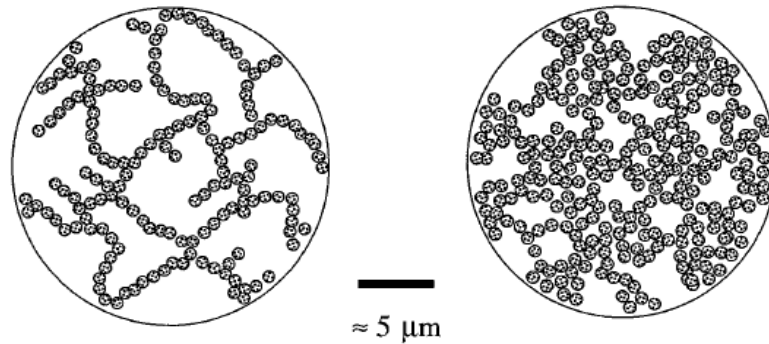
**PIV : coalescence de gouttelettes (Verdier & Brizard, 2002)**  
**t=0.24-1-1.48-4-6-8 s, PIB (36 Pa.s) dans PDMS (5 Pa.s), T=50°C**

# Techniques physiques particulières : Diffusion de rayonnements (X, neutrons, lumière)



- Diffusion des rayons donne informations sur la **microstructure** : taille, facteur de forme
- Analyse de la figure de diffusion
- Influence de l'écoulement

# Structure de la laponite (argile) sous écoulement (Pignon et al. 1996)



$D = 1$   
 $0.35\% \leq \phi_v \leq 0.48\%$

$D = 1.8$   
 $0.60\% \leq \phi_v \leq 2\%$

FIG. 2. Structure of Laponite according to volume fractions and fractal dimensions.

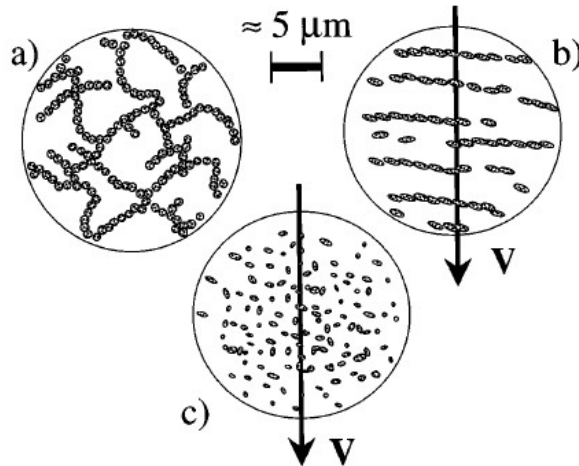
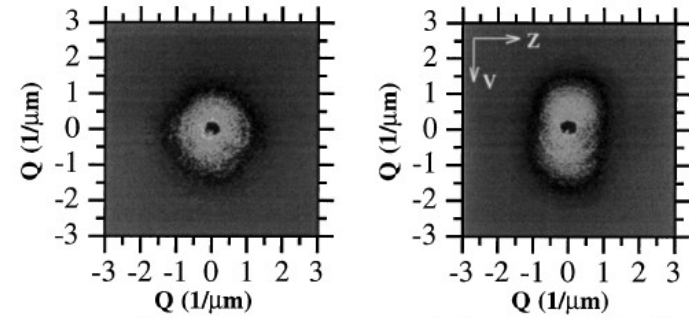
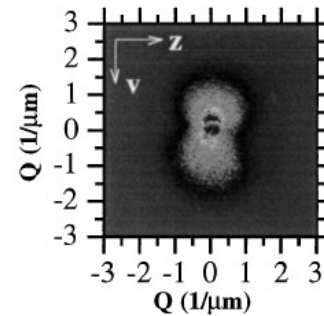


FIG. 2. Structure of Laponite (a) at rest (case  $D = 1$ ) (b) under shear below the critical shear rate, and (c) under shear beyond the critical shear rate.

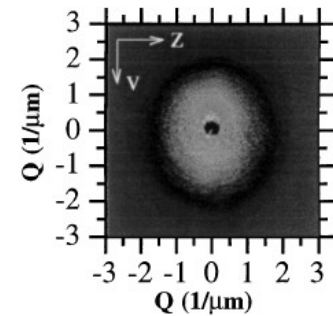


a1) At rest

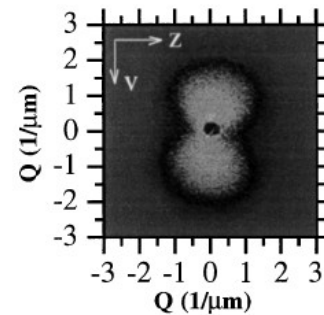
a4) Shear rate =  $100 \text{ s}^{-1}$



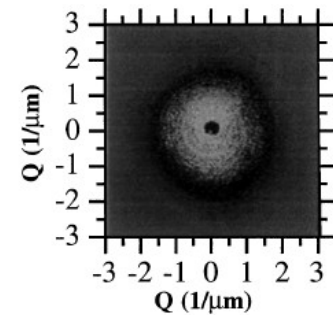
a2) Shear rate =  $10 \text{ s}^{-1}$



a5) Shear rate =  $180 \text{ s}^{-1}$



a3) Shear rate =  $40 \text{ s}^{-1}$




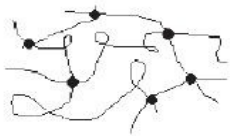
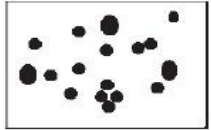



a6) at rest after shear

FIG. 3. SLS of suspension of Laponite under shear at  $\phi_v = 0.56\%$ ,  $[\text{NaCl}] = 10^{-3} \text{ mol/l}$ ,  $\text{pH} = 9.5$ ,  $t_p = 150 \text{ days}$ .

# Microstructure (Verdier, 2003)

TABLE I Rheological properties of a few complex

Systems	Applications (with some examples)	Microstructure and measuring techniques	Typical methods of characterization
<p><i>Polymers</i></p> 	<p><i>Melts</i></p> <ul style="list-style-type: none"> <li>Thermoplastics uncross-linked systems</li> </ul>	<ul style="list-style-type: none"> <li>Macromolecules entangled with each others</li> <li>Local friction + viscosity</li> <li>Polymer chains (<math>M_w</math>)</li> <li>Dilute system (<math>M_w &lt; M_c</math>)</li> <li>Entanglements (<math>M_w &gt; M_c</math>)</li> <li>Fluorescence (<math>M_c</math> = critical mass between weak crosslinks)</li> </ul>	<p><i>Shear</i></p> <ul style="list-style-type: none"> <li>Dynamic moduli <math>G', G''</math></li> <li>Viscosity <math>\eta \propto M_w^n</math> <math>n = 1 (M_w &lt; M_c)</math> <math>n = 3.4 (M_w &gt; M_c)</math></li> <li>Normal stresses</li> </ul> <p><i>Elongation:</i></p> <ul style="list-style-type: none"> <li>Strain hardening</li> <li>Elongational viscosity</li> <li>Time-temperature sup.</li> </ul>
	<p><i>Elastomers (cross-linked)</i></p>	<ul style="list-style-type: none"> <li>Network with strong cross-links (rubbers)</li> </ul>	<p><math>G', G''</math> like <math>\omega^n</math> (<math>n \approx 0.6</math>)</p>
	<p><i>Polymer solutions</i></p> <ul style="list-style-type: none"> <li>Polymers in a solvent (inks, ...)</li> </ul>	<ul style="list-style-type: none"> <li>Dilute solutions containing polymer chains (<math>c</math> = volume concentration)</li> </ul>	<p><math>[\eta] = \lim_{c \rightarrow 0} (\eta - \eta_s) / \eta_s c</math> (solvent viscosity = <math>\eta_s</math>)</p>
<p><i>Gels</i></p> 	<ul style="list-style-type: none"> <li>Physical gels</li> <li>Chemical gels</li> <li>Food products</li> <li>Pastes, slurries</li> <li>With polymers or particles</li> </ul>	<ul style="list-style-type: none"> <li>Network with cross-links (weak or strong gel)</li> <li>TEM (Transmission Electron Microscopy) + Light diffusion</li> </ul>	<ul style="list-style-type: none"> <li>Yield stress</li> <li>Elasticity (below yield) <math>\sigma = C_1 B + C_2 B^{-1}</math></li> <li>Microrheology <math>G', G''</math></li> <li><math>G' \approx G'' \approx \omega^n</math></li> </ul>
<p><i>Suspensions</i></p> 	<ul style="list-style-type: none"> <li>Micronic suspensions</li> <li>Paints, cosmetics</li> <li>Colloids, clays</li> <li>Blood</li> </ul>	<ul style="list-style-type: none"> <li>Particle size distribution (waves, diffusion)</li> <li>Shape of particles (rods.)</li> <li>Aggregates X-rays, Neutrons, Light</li> </ul>	<ul style="list-style-type: none"> <li>Non-Newtonian (shear thinning and thickening)</li> <li>Yield stress (fractal)</li> <li>Viscosity</li> <li>Diffusion</li> </ul>
<p><i>Binary systems</i></p> 	<ul style="list-style-type: none"> <li>Emulsions, blends</li> <li>Micellar solutions</li> <li>Foams</li> </ul>	<ul style="list-style-type: none"> <li>Tubes, plates or spheroids (phase inversion) TEM, SEM, Light microscopy</li> </ul>	<ul style="list-style-type: none"> <li><math>G' - G''</math></li> <li>Viscosity</li> <li>Ultrasound</li> </ul>

- microscopie optique
- confocale à balayage laser
- MEB
- TEM (transmission)
- diffusion (X, neutrons, Lumière)
- Ultrasons
- tomographie X

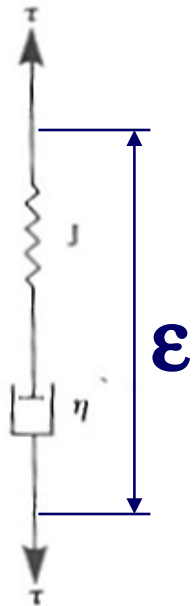


# Microstructure d'une suspension biologique (Mic optique)



$C_{\text{vol}} = 40-50 \%$  environ

# IV. Lois de comportement



**Maxwell : 1 mode**

**module :  $G = \eta_0 / \lambda = 1/J$**

**viscosité :  $\eta_0$**

**Temps de relaxation :  $\lambda$**

Equation différentielle

$$\sigma + \lambda \dot{\sigma} = \eta_0 \dot{\epsilon}$$

Forme intégrale :

$$\sigma(t) = \int_{-\infty}^t \eta_0 / \lambda \exp[(t' - t) / \lambda] \dot{\epsilon}(t') dt'$$

# Echelles de temps

$$\sigma + \lambda \dot{\sigma} = \eta_0 \dot{\varepsilon}$$

Maxwell

- si sollicitations rapides (temps courts) :  $t \ll \lambda$   
alors  $\lambda \dot{\sigma} \gg \sigma \implies \sigma \approx G \varepsilon$  ,  $G = \eta_0 / \lambda$

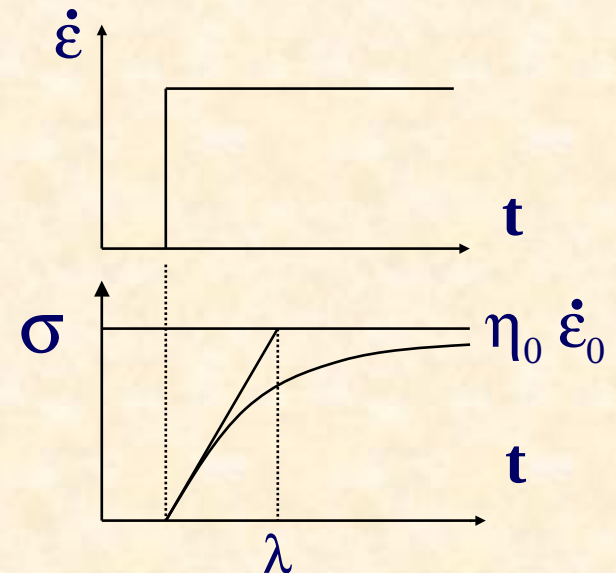
**comportement élastique**

- si sollicitations lentes (temps longs) :  $t \gg \lambda$   
alors  $\dot{\sigma} \approx 0 \implies \sigma \approx \eta_0 \dot{\varepsilon}$

**comportement visqueux**

NB : Réponse à un échelon :

$$\sigma(t) = \eta_0 \dot{\varepsilon}_0 (1 - \exp(-t/\lambda))$$



# Analyse des mesures dynamiques

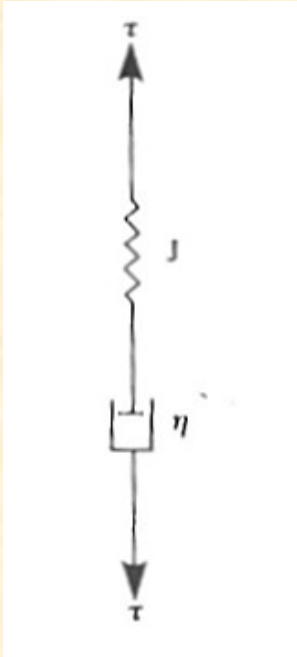
Maxwell : **1 mode**

**module**  $G = \eta_0 / \lambda = 1/J$

**viscosité**  $\eta_0$

**Temps de relax.**  $\lambda$

**Modèle de Maxwell :**  
**multimode ou continu**



$$G' = \frac{\eta_0 \omega^2 \lambda}{1 + \omega^2 \lambda^2}$$

$$\frac{G''}{\omega} = \eta' = \frac{\eta_0}{1 + \omega^2 \lambda^2}$$

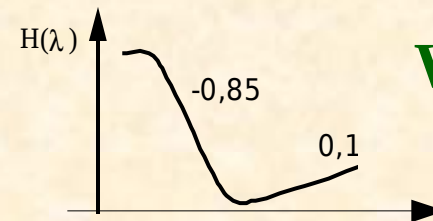
$$\tan \delta = \frac{1}{\lambda \omega}$$

$$G' = \int_0^{\infty} \frac{\omega^2 \lambda^2 H(\lambda)}{1 + \omega^2 \lambda^2} d \ln \lambda$$

$$\eta' = \frac{G''}{\omega} = \int_0^{\infty} \frac{H(\lambda) d\lambda}{1 + \omega^2 \lambda^2}$$

**1D**

$$\sigma + \lambda \dot{\sigma} = \eta_0 \dot{\epsilon} \Rightarrow \sigma(1 + i\lambda\omega) = \eta_0 i\omega\epsilon$$



**Winter (1997)**

$$H(\lambda) = H_e \lambda^{ne} + H_g \lambda^{-ng} \quad \lambda < \lambda_{\max}$$

# Modèles élastiques et visqueux (3D)

## Elasticité

$$\boldsymbol{\tau} = G\mathbf{B} \quad \text{or} \quad \mathbf{T} = -p\mathbf{I} + G\mathbf{B}$$

## Modèle élastique généralisé

$$\mathbf{T} = -p\mathbf{I} + g_1(I_B, II_B)\mathbf{B} + g_2(I_B, II_B)\mathbf{B}^{-1}$$

## Viscosité

$$\mathbf{T} = -p\mathbf{I} + \eta 2\mathbf{D}$$

## Visqueux généralisé

$$\mathbf{T} = -p\mathbf{I} + \eta_1 2\mathbf{D} + \eta_2 (2\mathbf{D})^2$$

# Modèles viscoélastiques

- Fluide du 2<sup>nd</sup> ordre

$$\mathbf{T} = -p\mathbf{I} + 2\eta_0\mathbf{D} - \psi_{1,0}\overset{\nabla}{\mathbf{D}} + 4\psi_{2,0}\mathbf{D} \cdot \mathbf{D}$$

Dérivée convectée  
supérieure

$$\overset{\nabla}{\mathbf{A}} \equiv \dot{\mathbf{A}} - (\nabla\mathbf{v})^T \cdot \mathbf{A} - \mathbf{A} \cdot \nabla\mathbf{v}$$

$$\dot{\mathbf{A}} \equiv \frac{\partial}{\partial t} \mathbf{A} + \mathbf{v} \cdot \nabla \mathbf{A}$$

- Modèle de Maxwell

$$\boldsymbol{\tau} + \lambda \overset{\nabla}{\boldsymbol{\tau}} = 2\eta_0\mathbf{D}$$

- Modèle de Lodge

$$\boldsymbol{\tau} = \int_{-\infty}^t \frac{\eta_0}{\lambda^2} e^{-(t-t')/\lambda} (\mathbf{B}(t, t') - \mathbf{I}) dt'$$

# Modèles intégraux

$$\tau = \int_{-\infty}^t M(t-t') \left[ \phi_1(I_B, II_B) \mathbf{B}(t, t') + \phi_2(I_B, II_B) \mathbf{B}^{-1}(t, t') \right] dt'$$

TABLE 4.4.1 / Kernel Functions  $\phi_1$  and  $\phi_2$  for Superposition Integral Equations

Authors	$\phi_1$	$\phi_2$	Fits to Data for Polymer Melts
Wagner et al. (1979)	$f_1 e^{n_1 \sqrt{I-3}} + f_2 e^{-n_2 \sqrt{I-3}};$ $I = \alpha I_B + (1-\alpha) II_B$	0	For IUPAC A polyethylene, $f_1 = 0.57; f_2 = 0.43;$ $n_1 = 0.31; n_2 = 0.106;$ $\alpha = 0.032$
Papanastasiou, et al. (1983)	$\frac{1}{1+a(I_B-3)+b(II_B-3)}$	0	For IUPAC A polyethylene, $a = 0.0013; b = 0.068$ For a polystyrene, $a = 0.0021; b = 0.093$ For a polydimethylsiloxane, $a = 0.0040; b = 0.195$
Wagner and Demarmels (1990)	$\frac{1+\beta}{1+a\sqrt{(I_B-3)(II_B-3)}}$	$\frac{\beta}{1+a\sqrt{(I_B-3)(II_B-3)}}$	For Meissner's polyisobutylene, $a = 0.11; \beta = -0.27$
Doi and Edwards (1978); also see Currie (1980)	$\frac{5}{I-1};$	$\frac{-5}{(I-1)\sqrt{II_B+13/4}}$	K-BKZ kernel function <sup>a</sup> $U = \frac{5}{2} \ln[(I-1)/7]$ where $I = I_B + 2\sqrt{II_B+13/4}$

<sup>a</sup>This potential is based on a molecular theory; there are no adjustable parameters. Good fits are obtained only with polymers without long-chain branching for which the polydispersity is not too high.

Macosko,  
1994

# Modèles différentiels

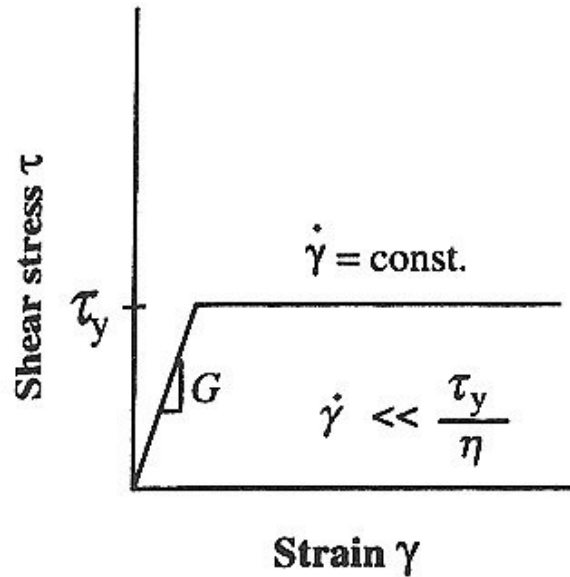
$$\nabla \tau + f_c(\tau, \mathbf{D}) + \frac{1}{\lambda} \tau + f_d(\tau) = 2G\mathbf{D}$$

Authors	Constitutive Models		Fits to Data for Polymer Melts
	$f_c$	$f_d$	
Johnson and Segalman (1977)	$\xi(\mathbf{D} \cdot \boldsymbol{\tau} + \boldsymbol{\tau} \cdot \mathbf{D})$	0	Predicts negative shear stress in step shear. Spurious oscillations in start-up of steady shearing. Singularities in steady extensional flows.
White and Metzner (1963, 1977)	$a(2\mathbf{D}:\mathbf{D})^{1/2}$	0	Poor fits in step shears. $N_2 = 0$ . Singularities in steady extensional flows.
Larson (1984)	$\frac{2\alpha}{3G} \mathbf{D}:\boldsymbol{\tau}(\boldsymbol{\tau} + G\mathbf{I})$	0	Fits data reasonably well for a variety of different types of deformation, except it predicts $N_2 = 0$
Giesekus (1966, 1982)	0	$\frac{\alpha}{\lambda G} \boldsymbol{\tau} \cdot \boldsymbol{\tau}$	Excellent fits in shearing flows; not the best for extensional flows.
Leonov (1976)	0	$\frac{\boldsymbol{\tau} \cdot \boldsymbol{\tau}}{2G\lambda} - \frac{(\boldsymbol{\tau} + \mathbf{I})}{6G\lambda} \text{tr}(\boldsymbol{\tau} + \mathbf{I}) + \frac{G(\boldsymbol{\tau} + \mathbf{I})}{6\lambda} \text{tr}(\boldsymbol{\tau} + \mathbf{I})^{-1}$	Excellent fits in shearing flows; not the best for extensional flows. No fitting parameters other than those of linear viscoelasticity.
Phan Thien and Tanner (1977, 1978)	$\xi(\mathbf{D} \cdot \boldsymbol{\tau} + \boldsymbol{\tau} \cdot \mathbf{D})$	$\frac{1}{\lambda} \exp\left(\frac{\beta}{G} \text{tr} \boldsymbol{\tau}\right)(\boldsymbol{\tau} - \mathbf{I})$	Fits data reasonably well for a variety of different types of deformation. But there are spurious oscillations in start-up of steady shearing when $\xi \neq 0$ ; and when $\xi = 0$ , then $N_2 = 0$ .

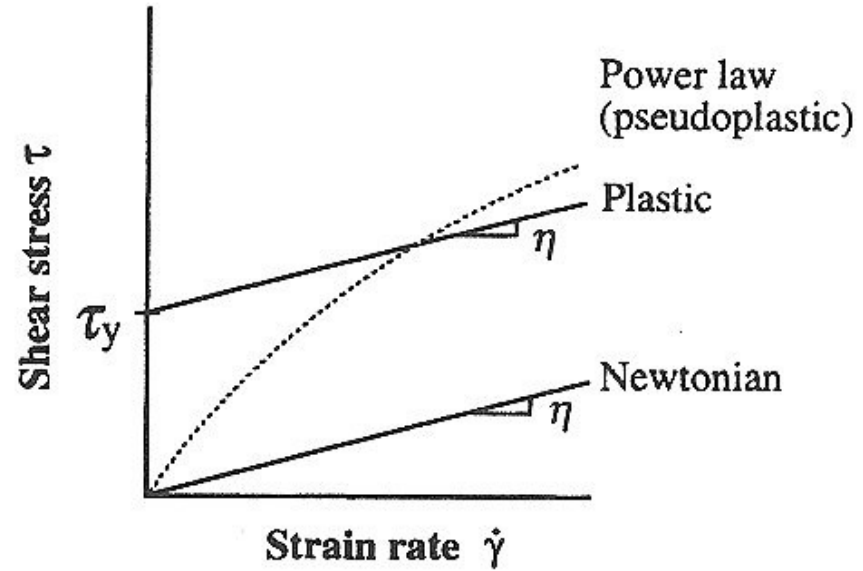
**Macosko,  
1994**



# Modèles de fluides à seuil



(a)



(b)

$$\tau = G\mathbf{B} \quad \text{for} \quad II_{\tau} < \tau_y^2$$

## Modèle de Bingham

$$\tau = \left[ \eta_0 + \frac{\tau_y}{|II_{2D}|^{1/2}} \right] 2\mathbf{D} \quad \text{for} \quad II_{\tau} \geq \tau_y^2$$

## Modèle de Herschel-Bulkley

$$\tau = 2\eta\mathbf{D} \quad \text{for} \quad II_{2D}^{1/2} \leq \dot{\gamma}_c$$

$$\tau = 2 \left[ \frac{\tau_y}{|II_{2D}|^{1/2}} + m|II_{2D}|^{(n-1)/2} \right] \mathbf{D} \quad \text{for} \quad II_{2D}^{1/2} > \dot{\gamma}_c$$

# Conclusions:

Rhéologie = science très complète

