

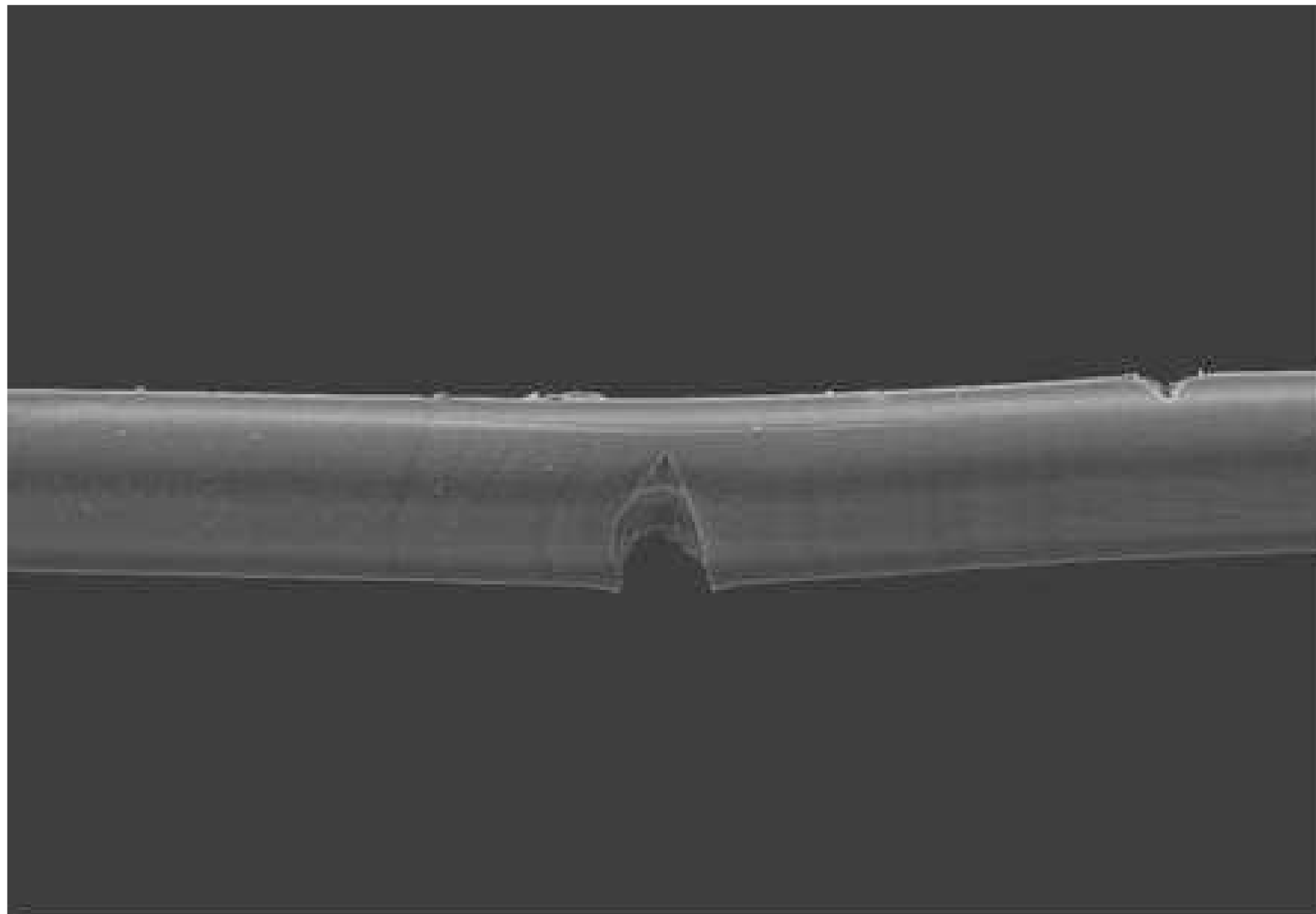
Expertise des ruptures dans des structures renforcées par des fibres

A.R.Bunsell

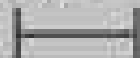
Mines Paristech, Centre des Matériaux

<anthony.bunsell@ensmp.fr>

Cordes de parachute en nylon



10µm



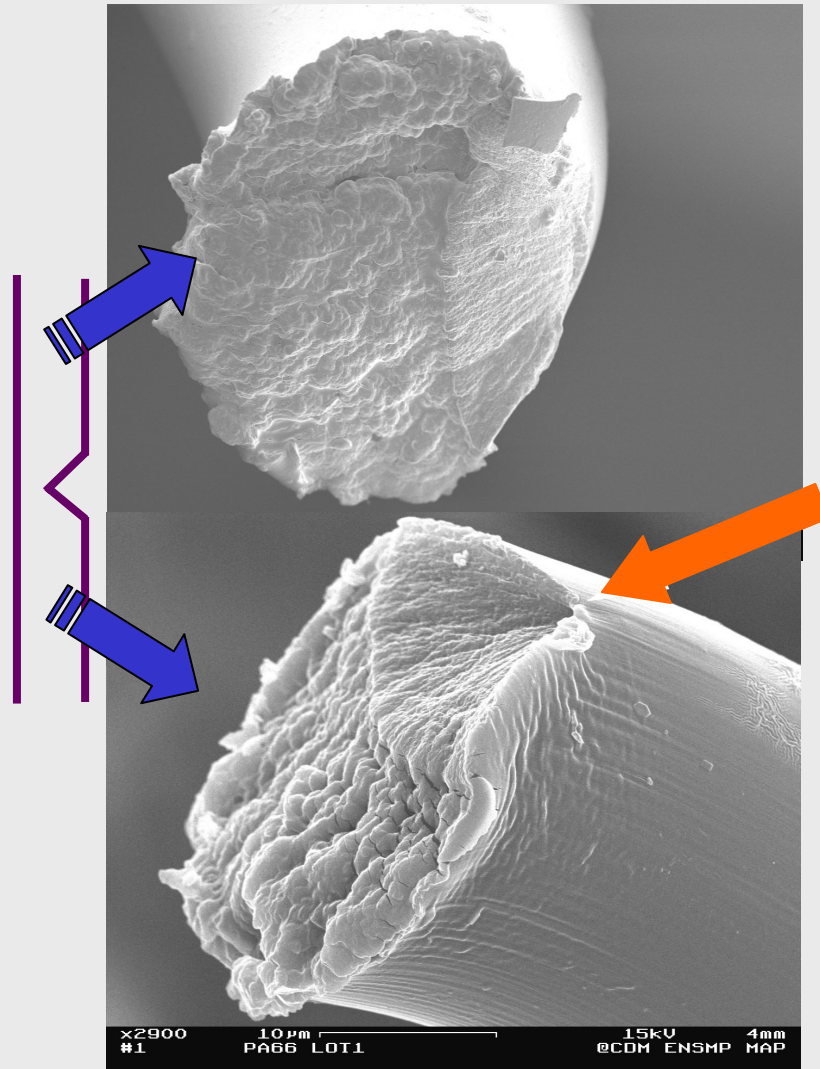
Grain = 709°C

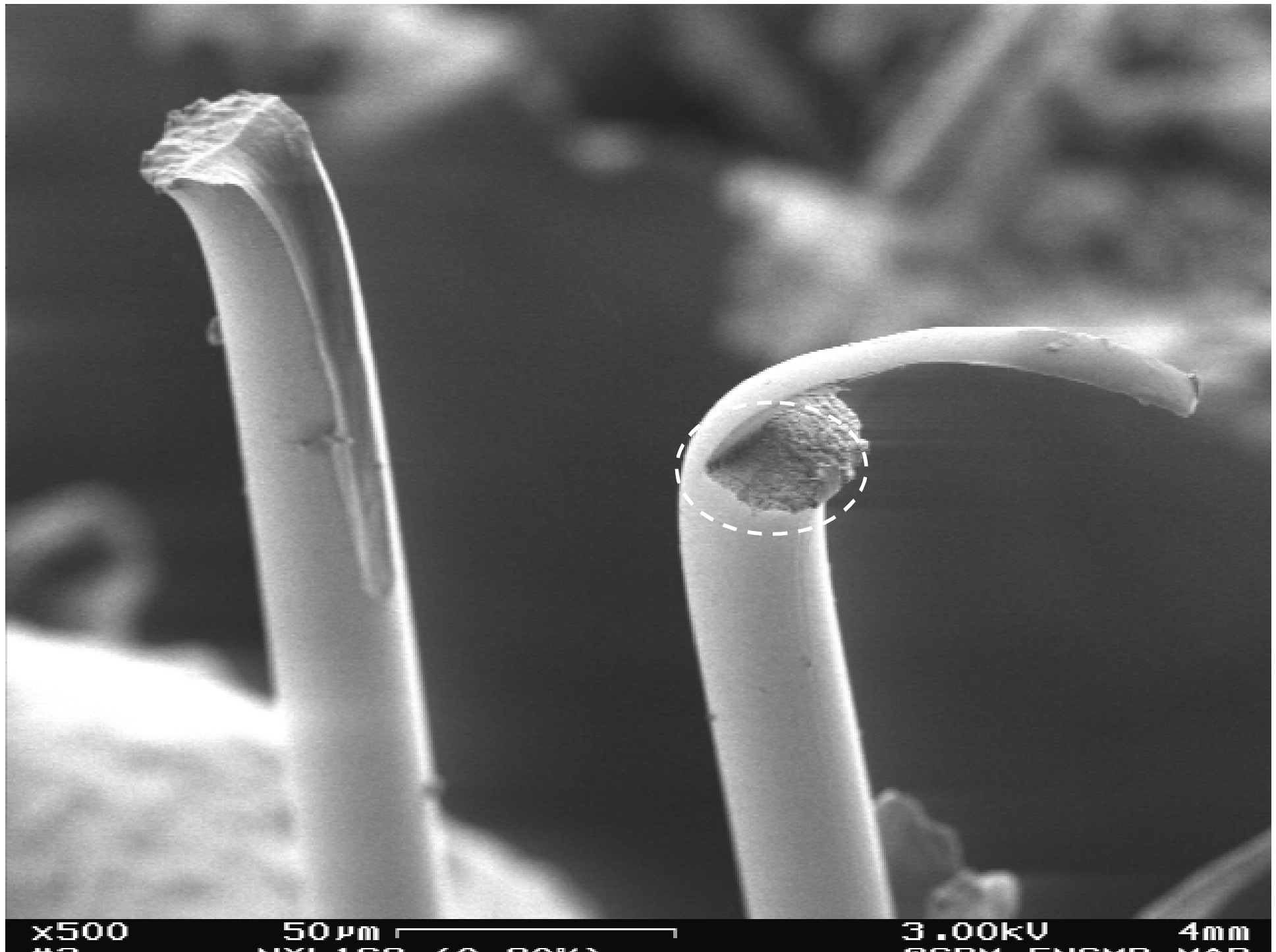
EHT = 10.00 kV WD = 14 mm

MAP @CDSIENSMP

Signal A = SE1

Facies de rupture en traction des fibres de polyamide 66



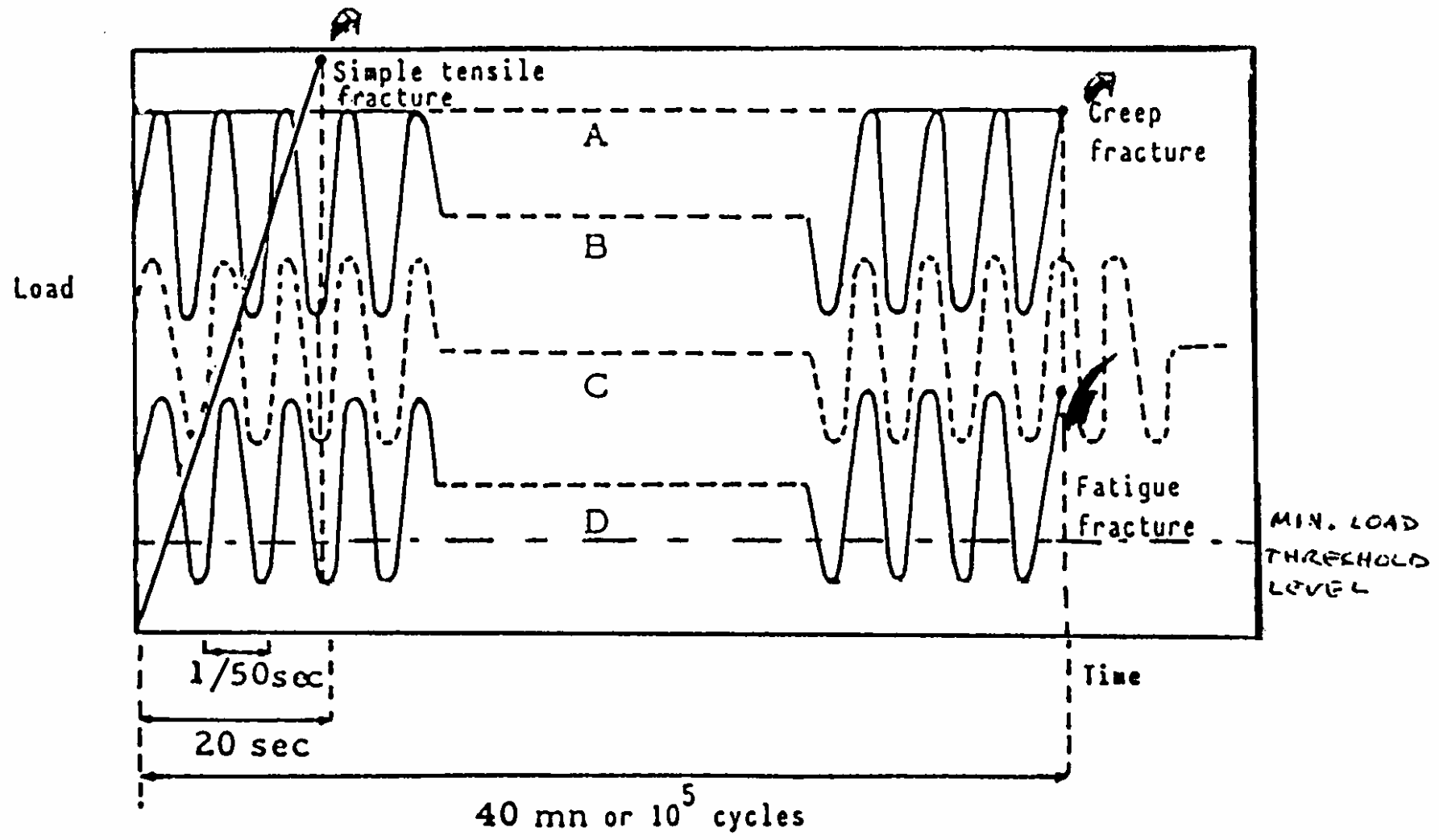


x500

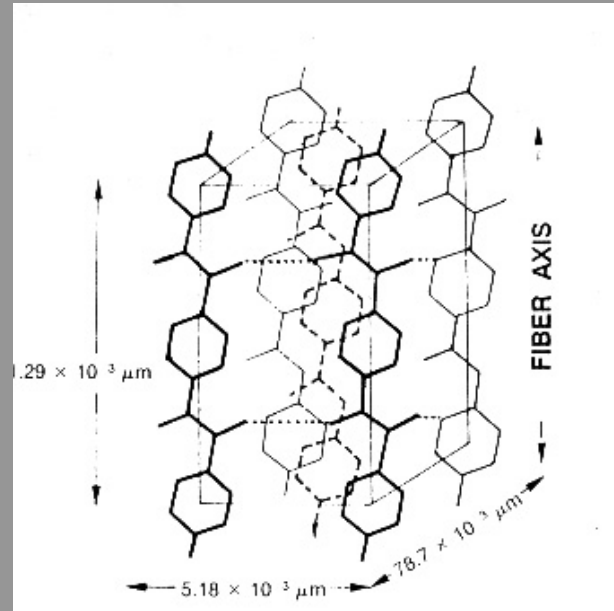
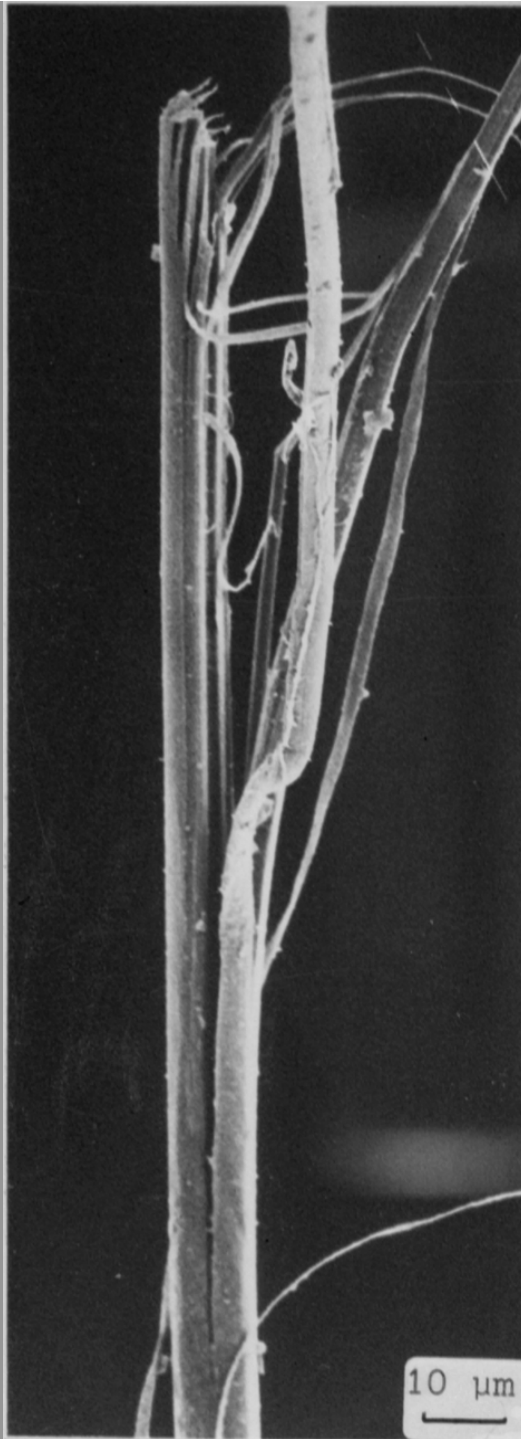
50µm

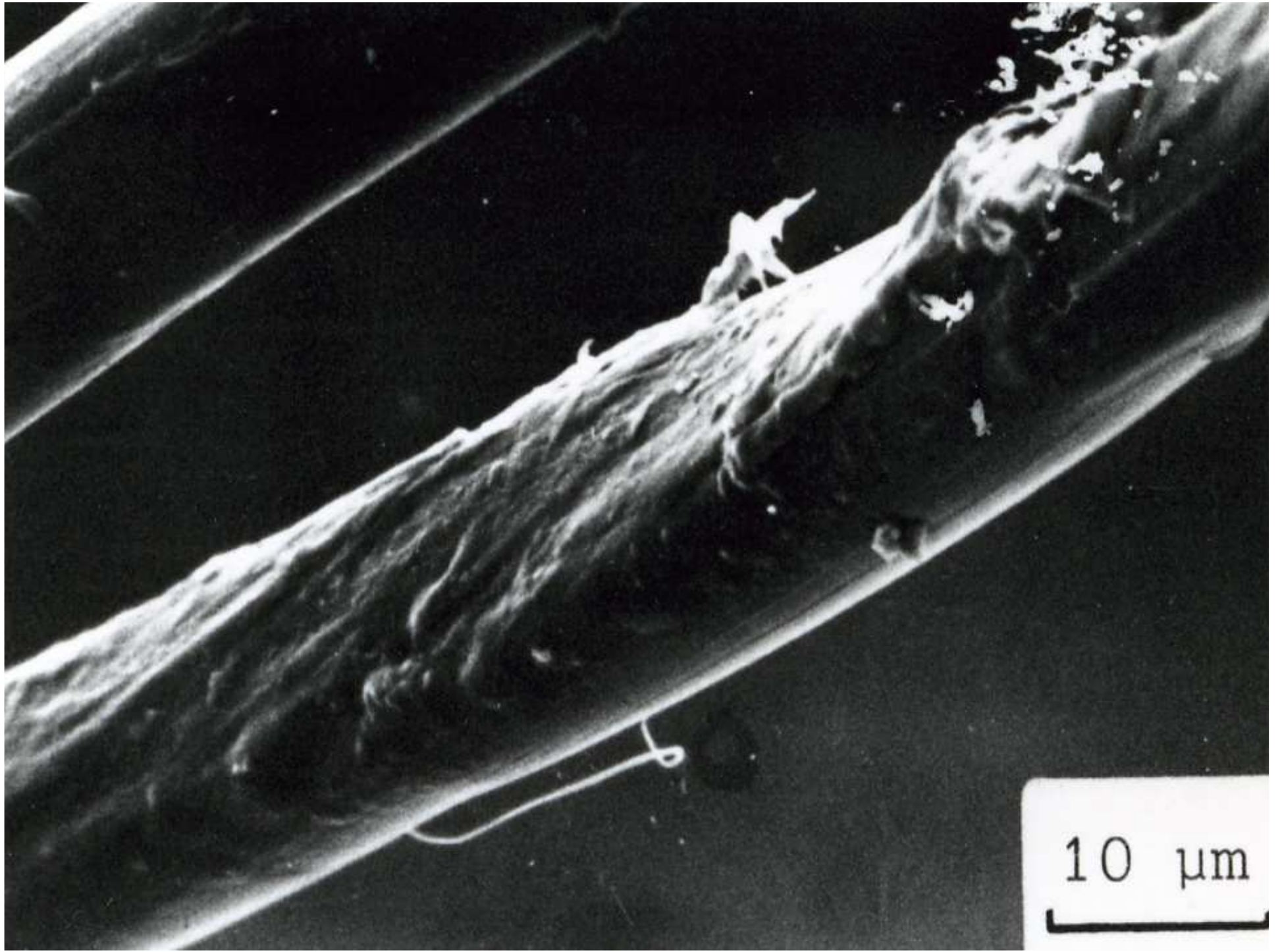
3.00kV

4mm

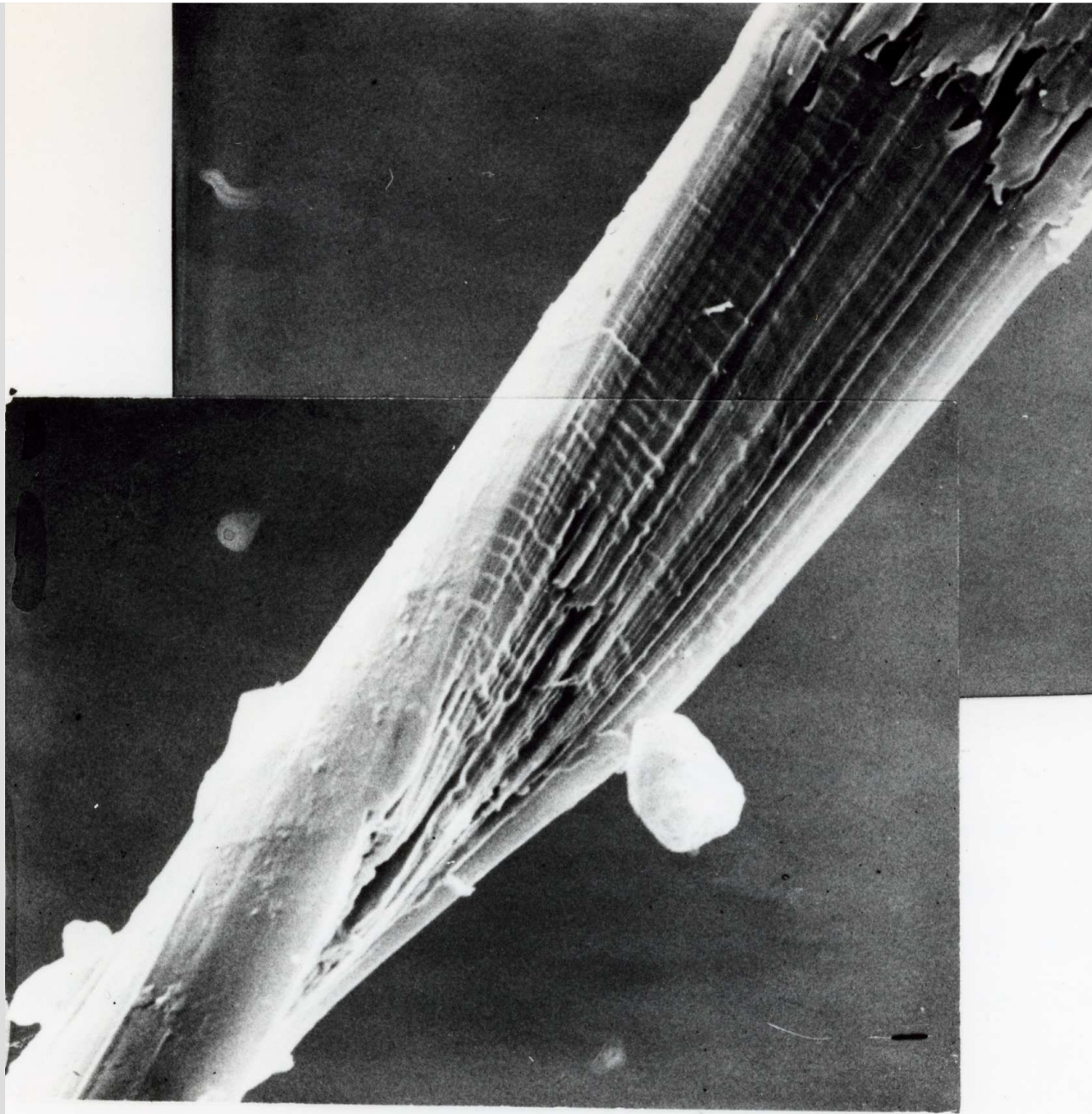


Rupture des câbles de remorquage en Kevlar

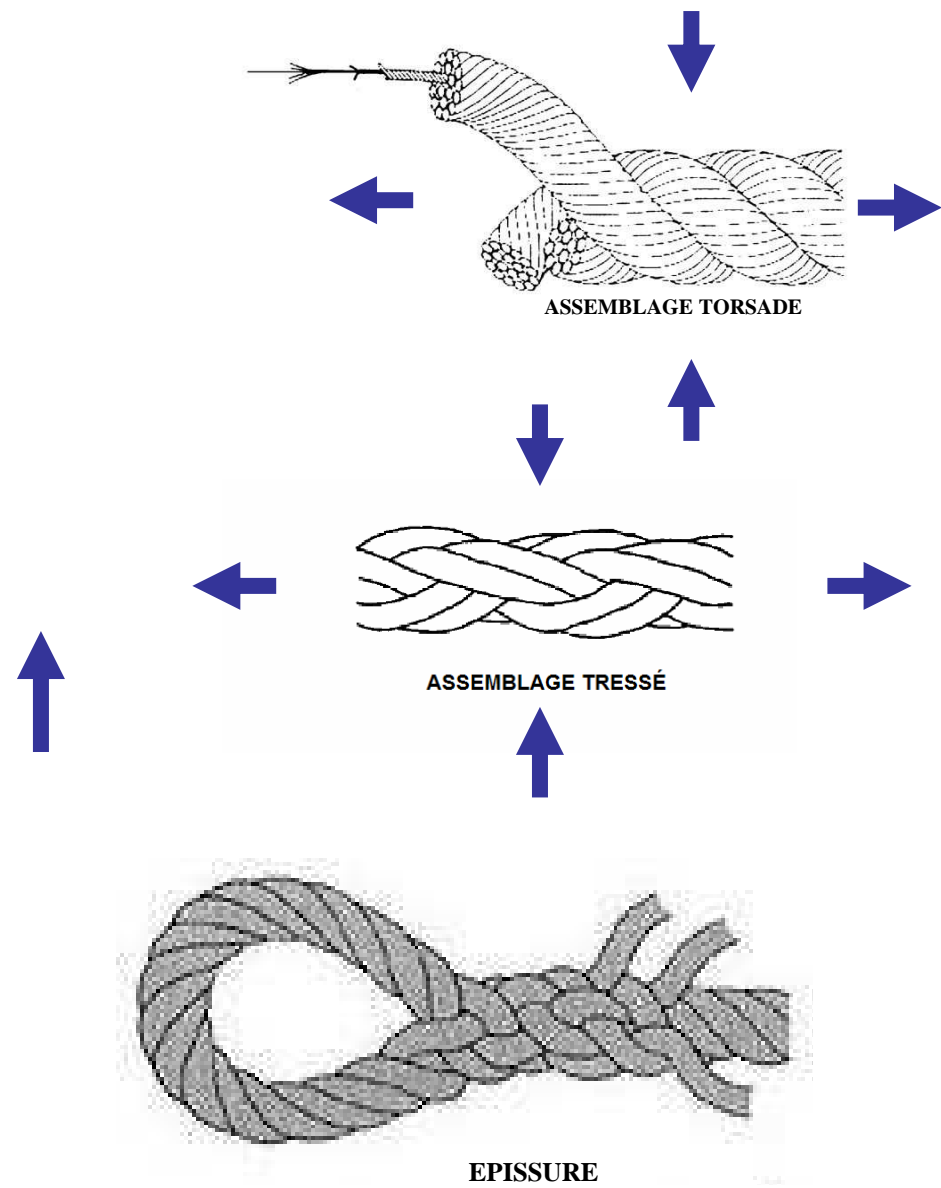


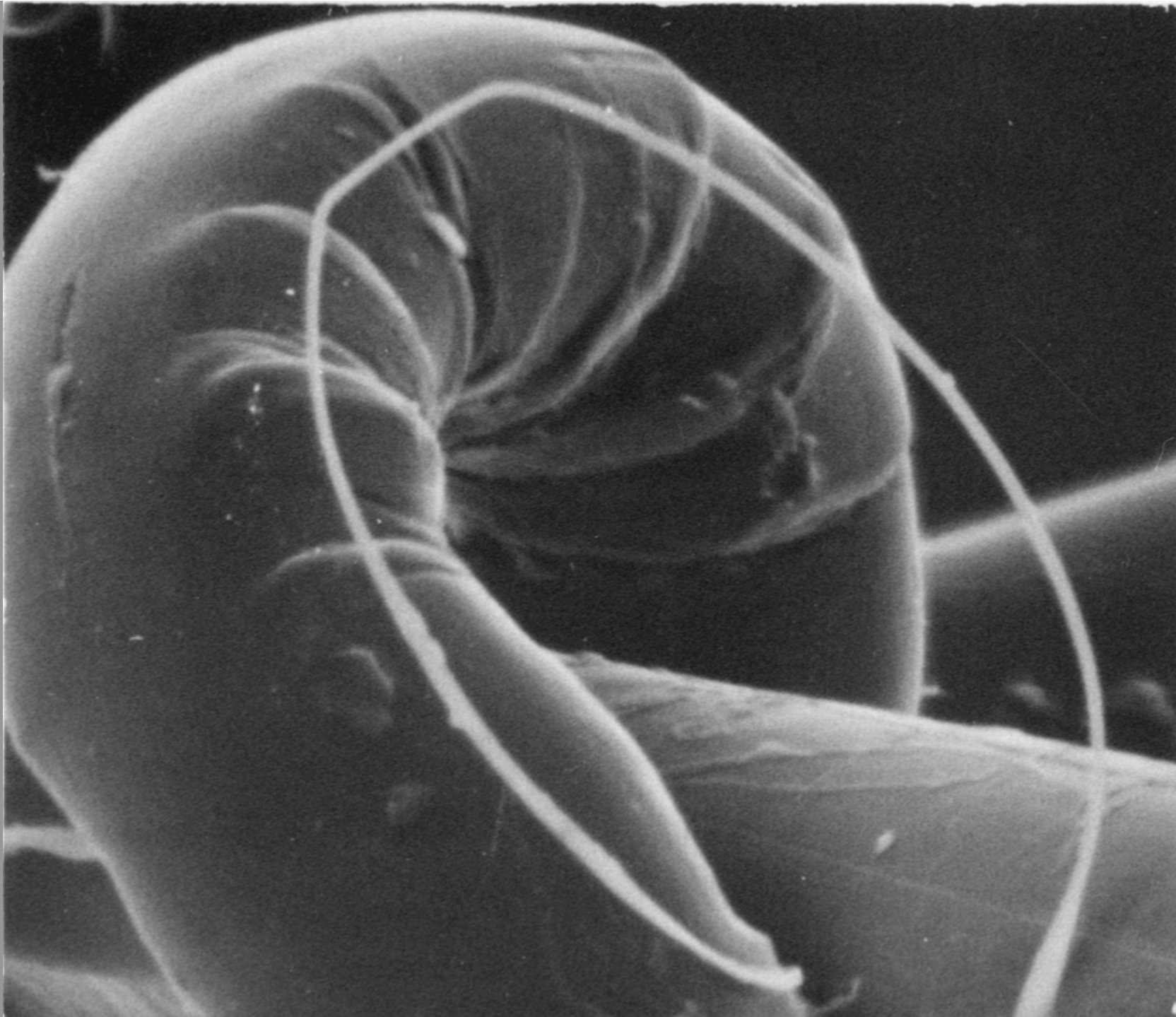


10 μm

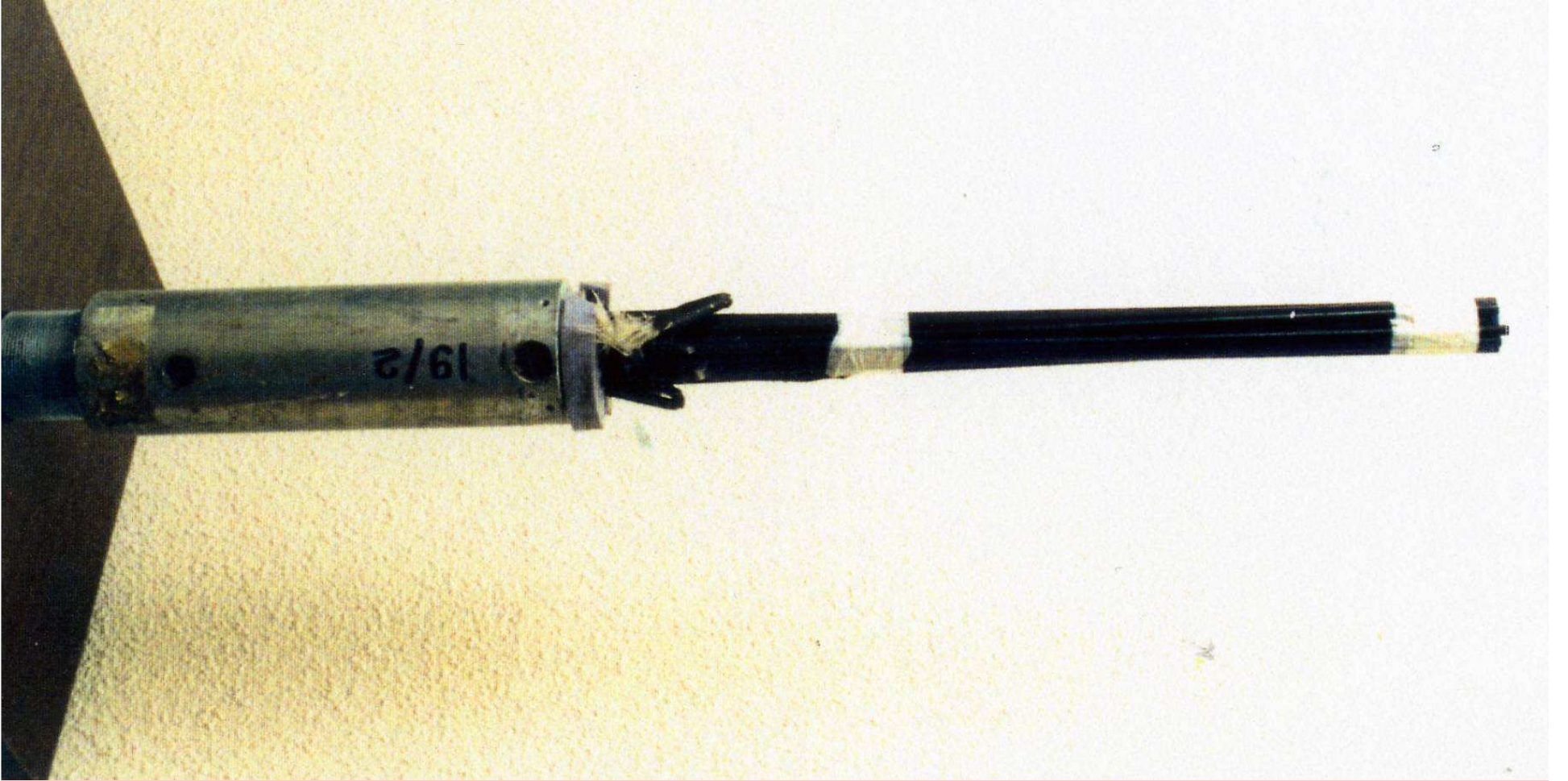


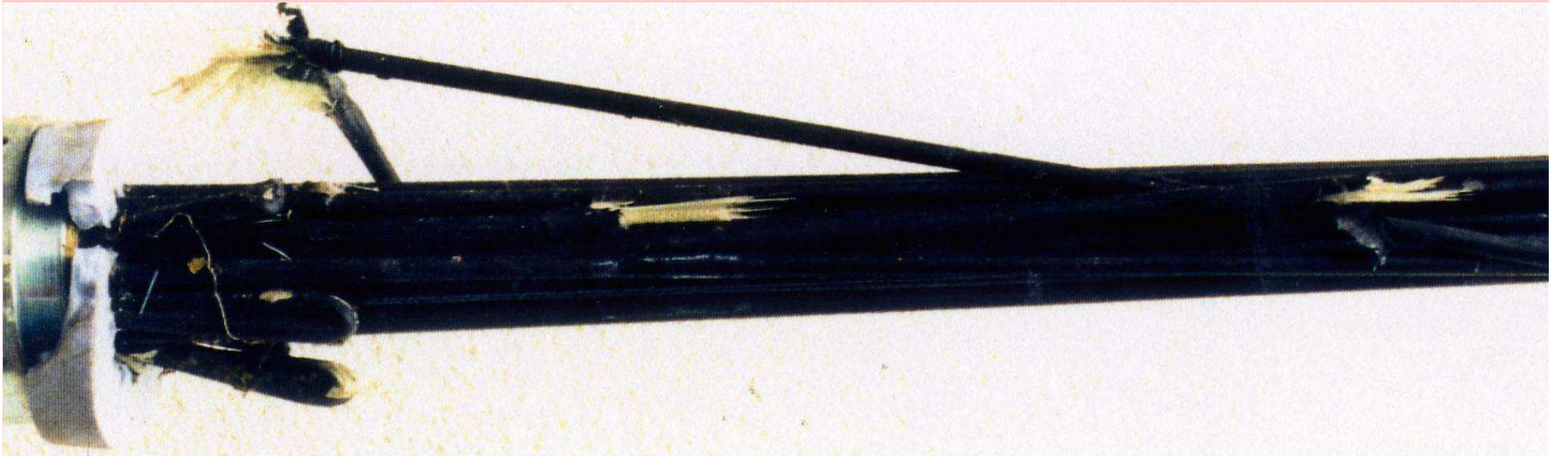
10 μm

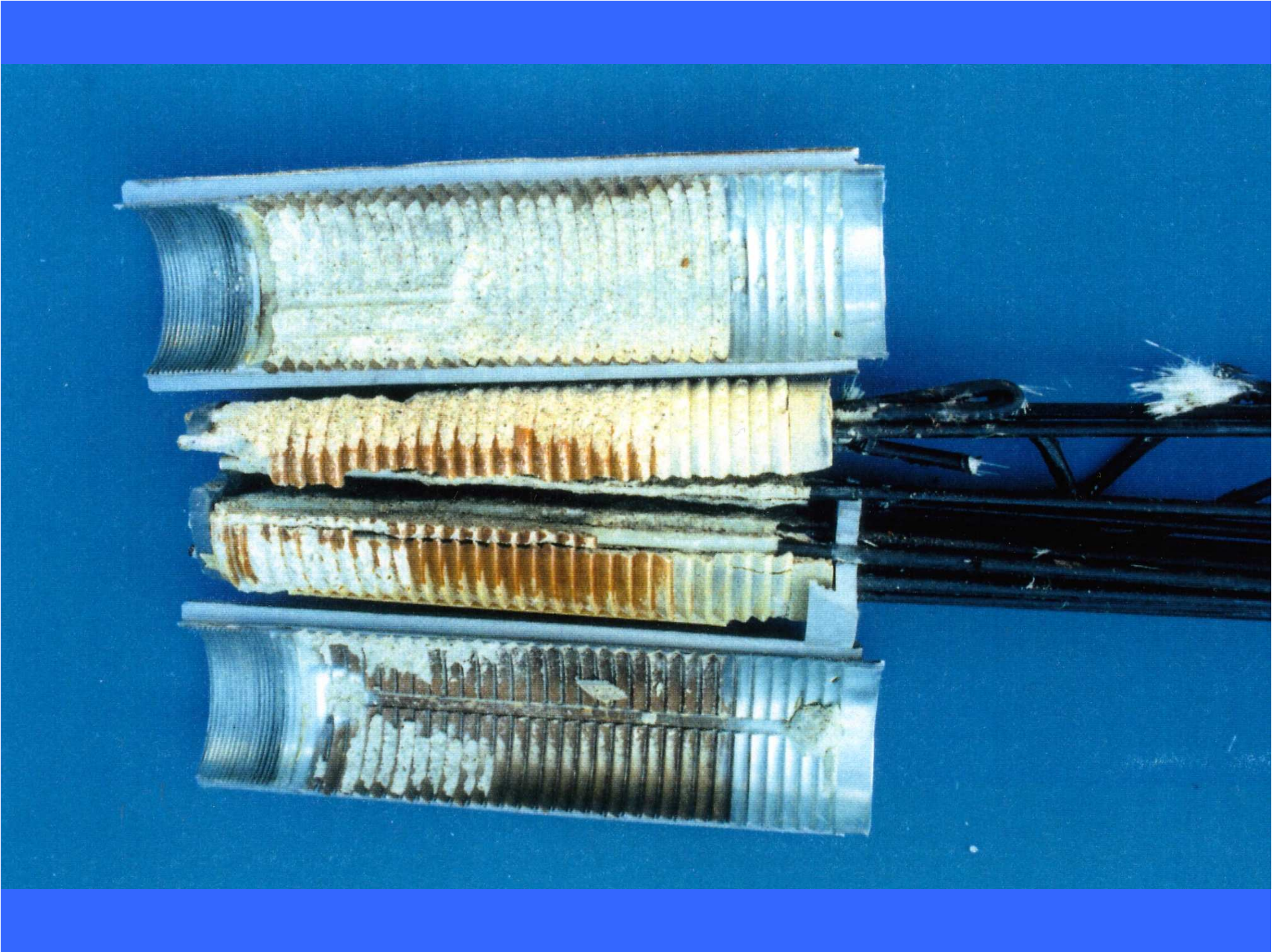


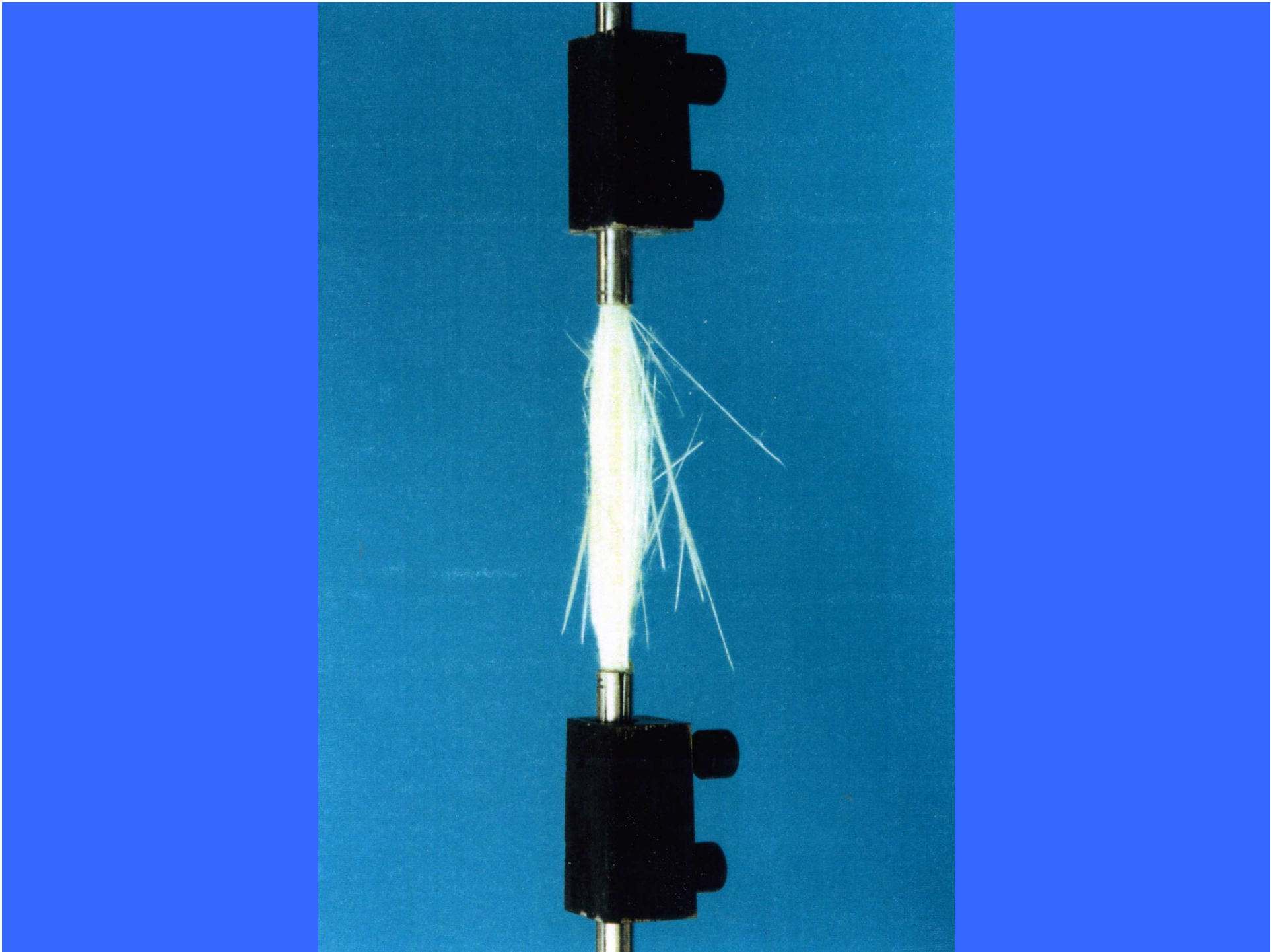


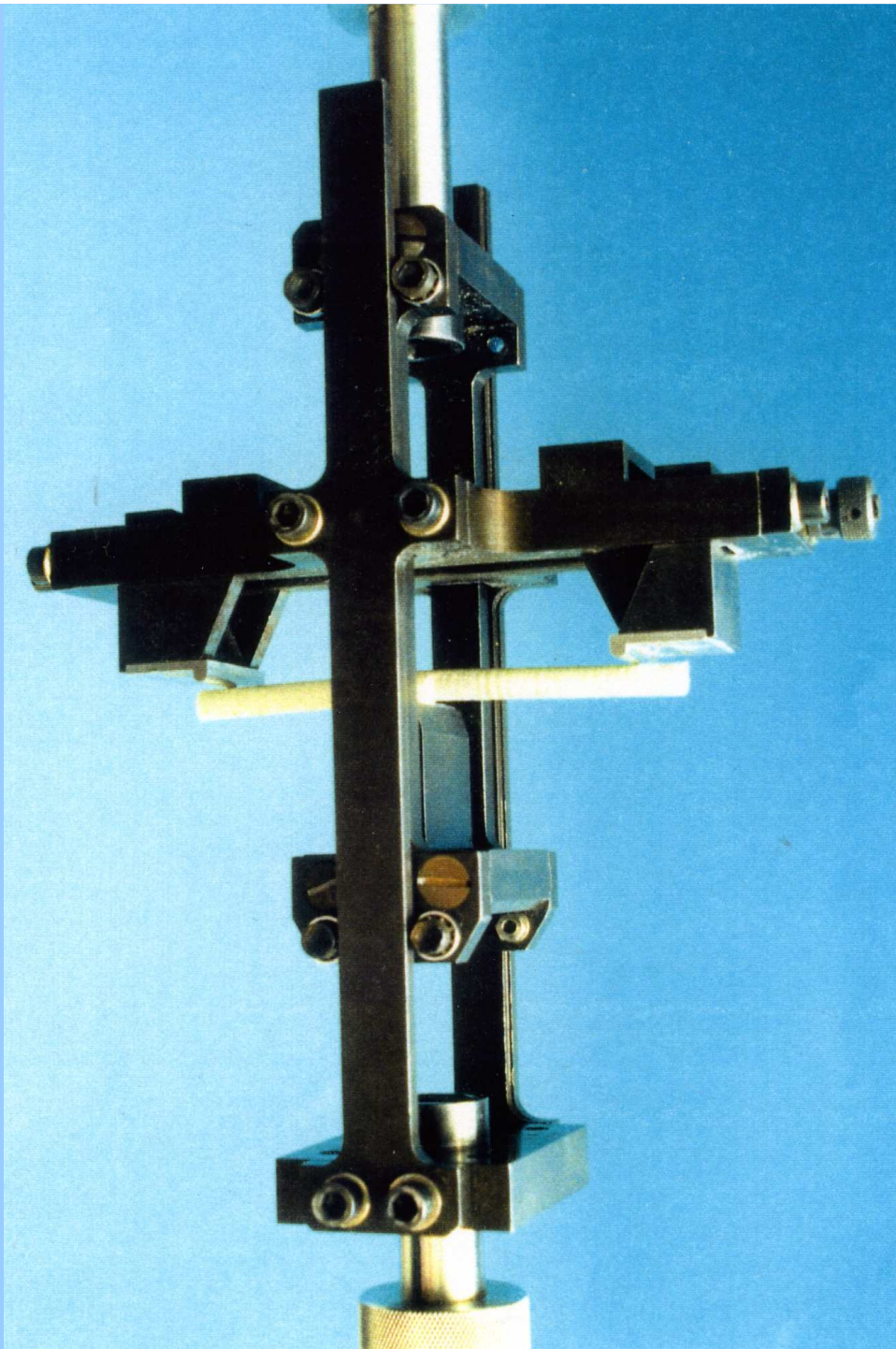
Rupture des joncs de tirants



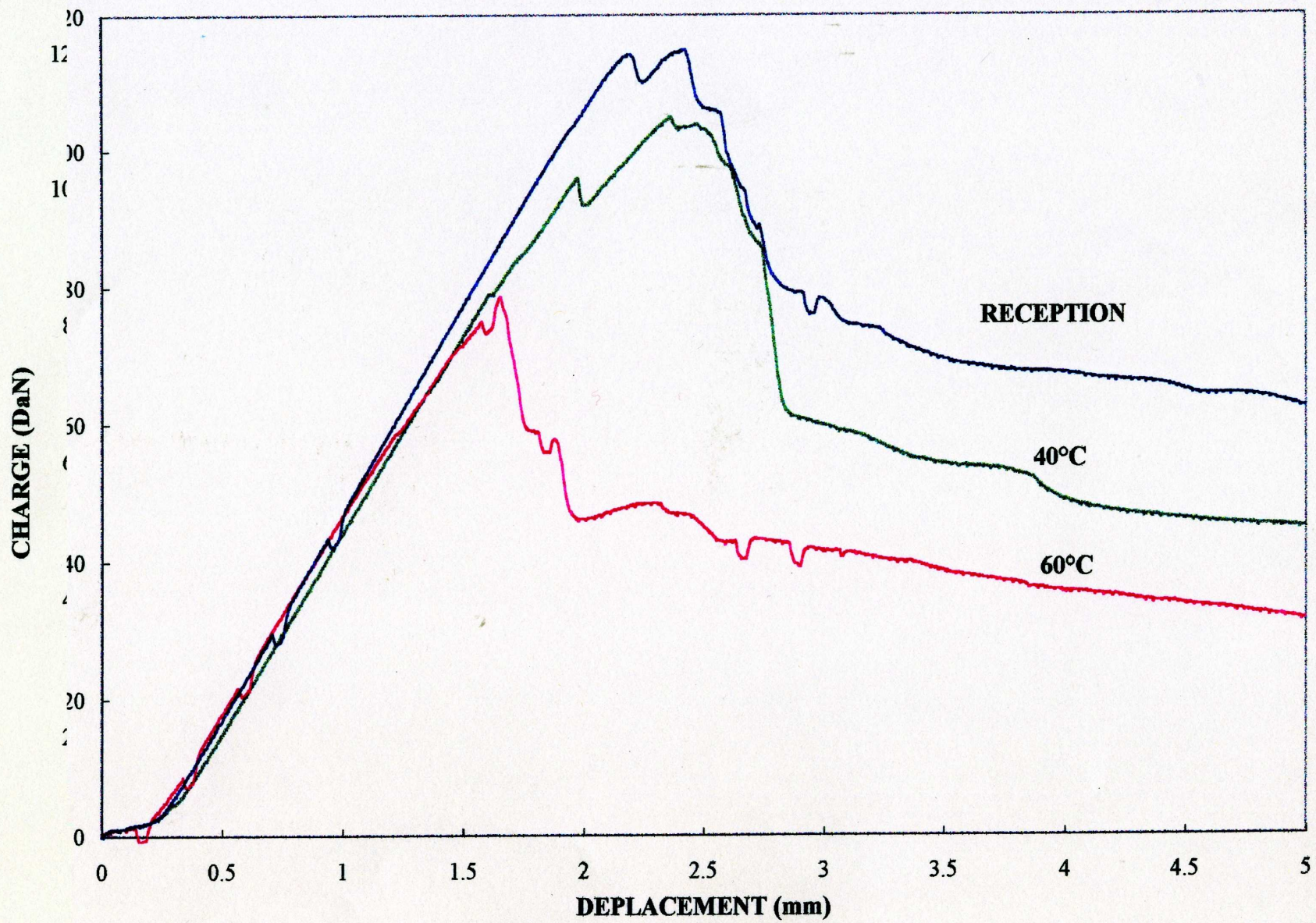




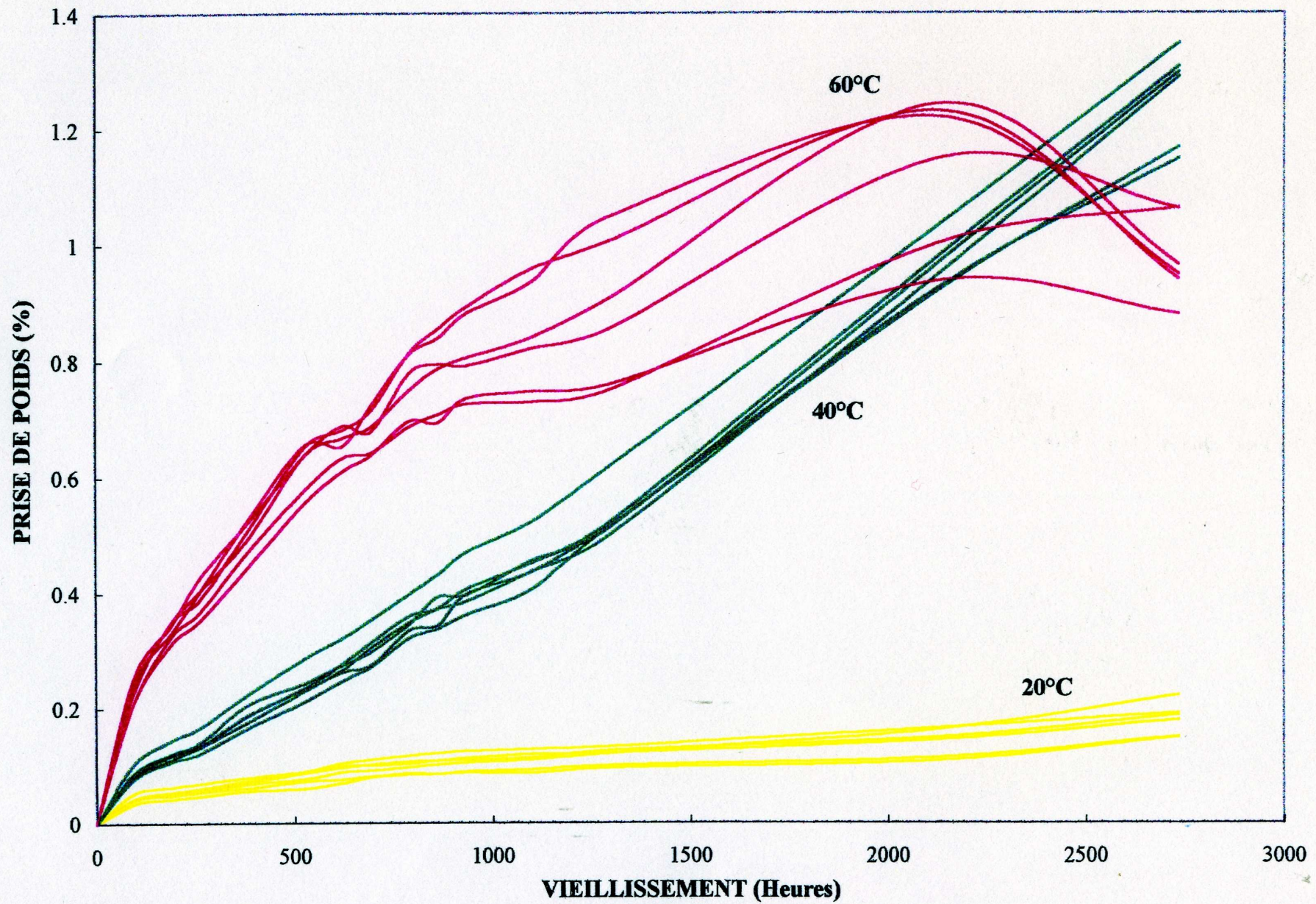




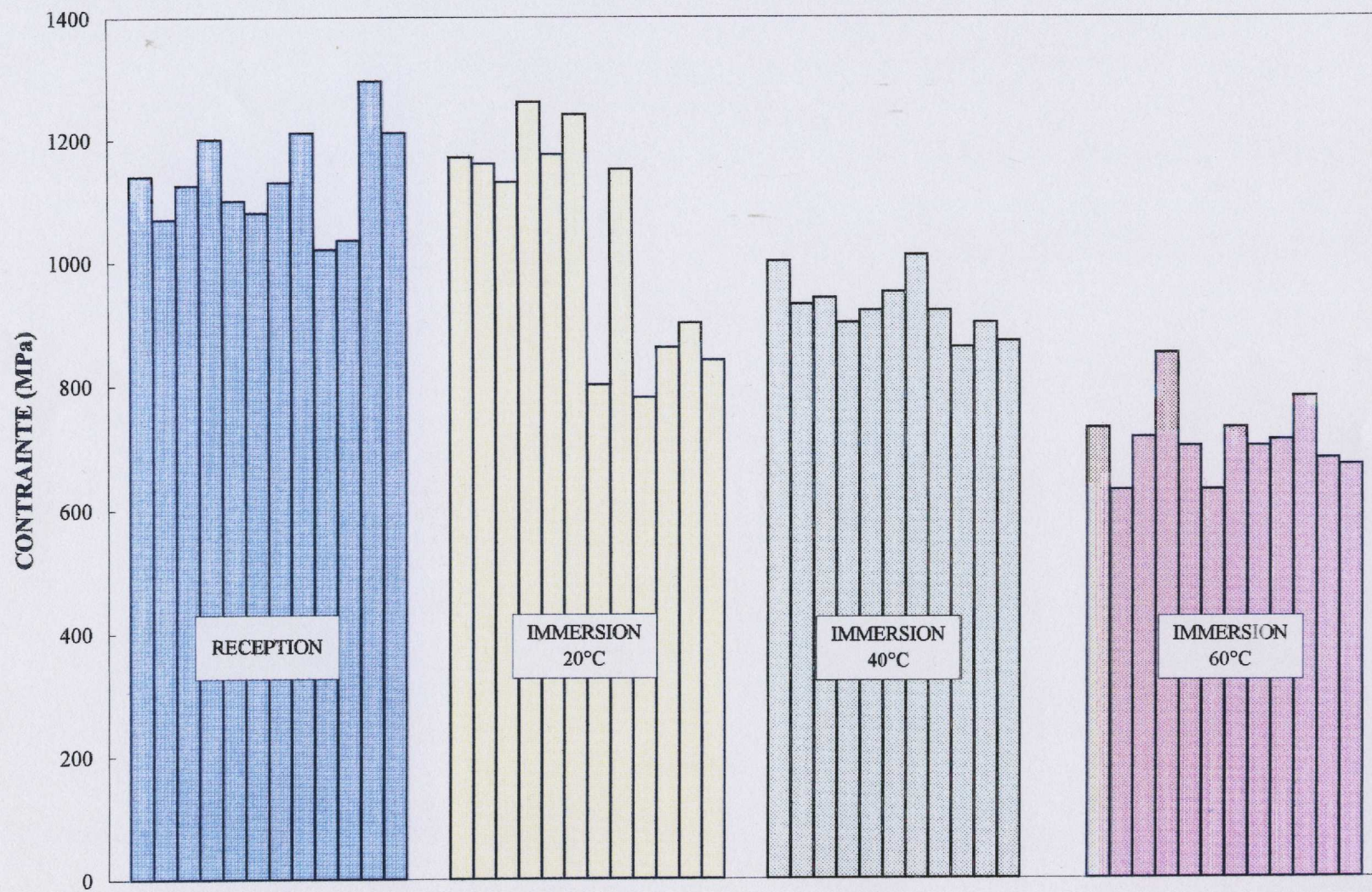




Comparaison du comportement du matériau en flexion

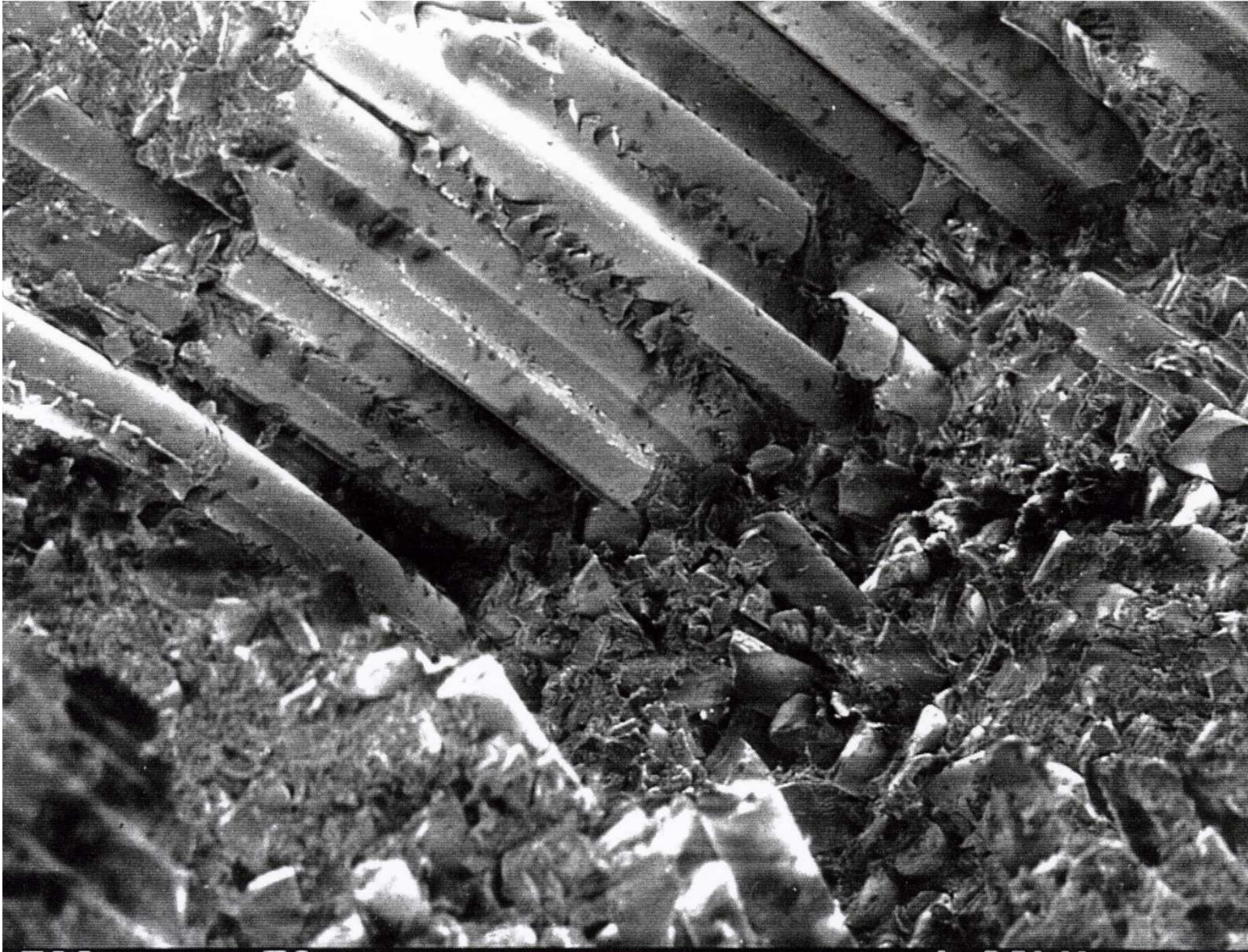


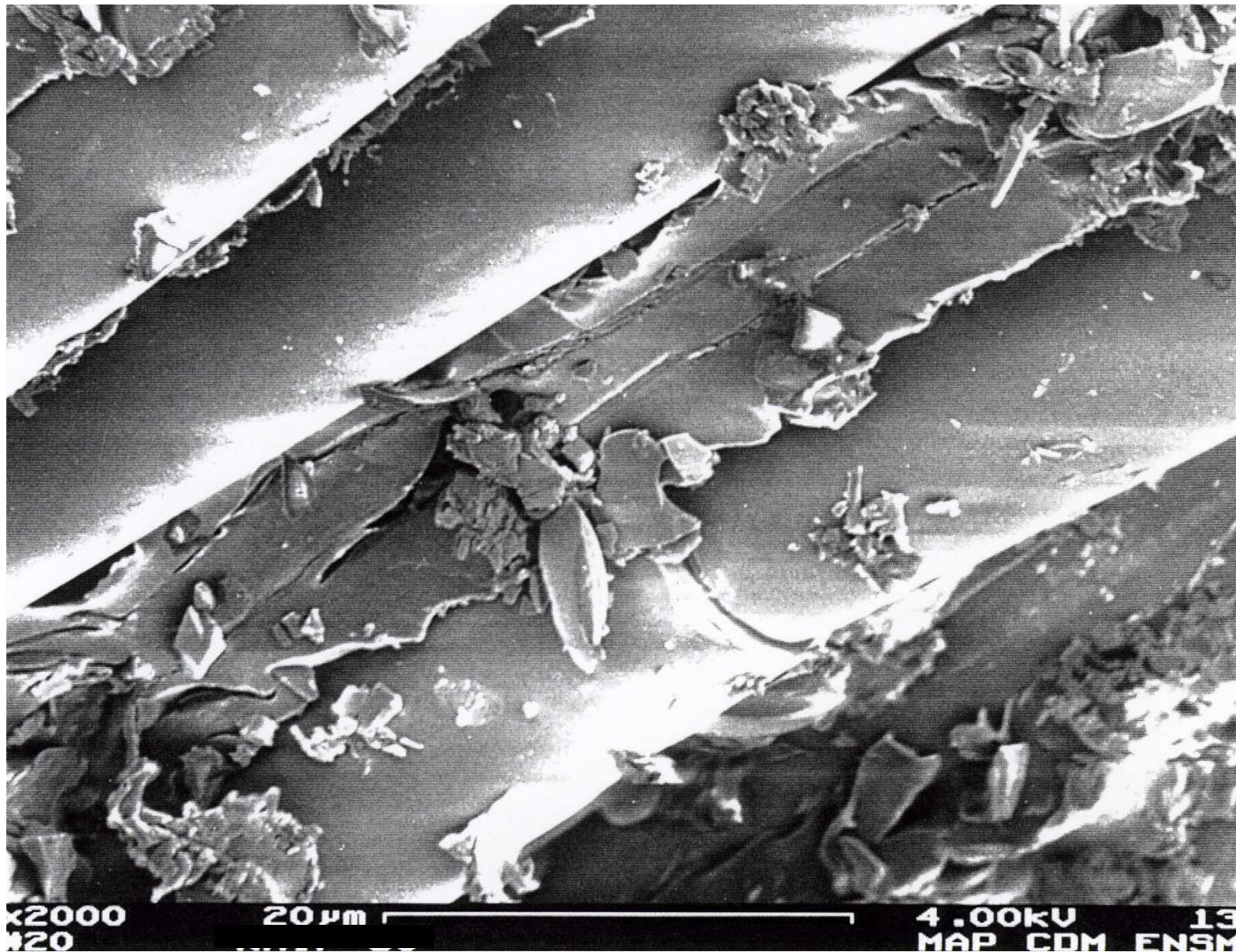
Comparaison de la prise de poids du matériau en immersion à différentes températures



Contrainte à la rupture en flexion à différents vieillissements



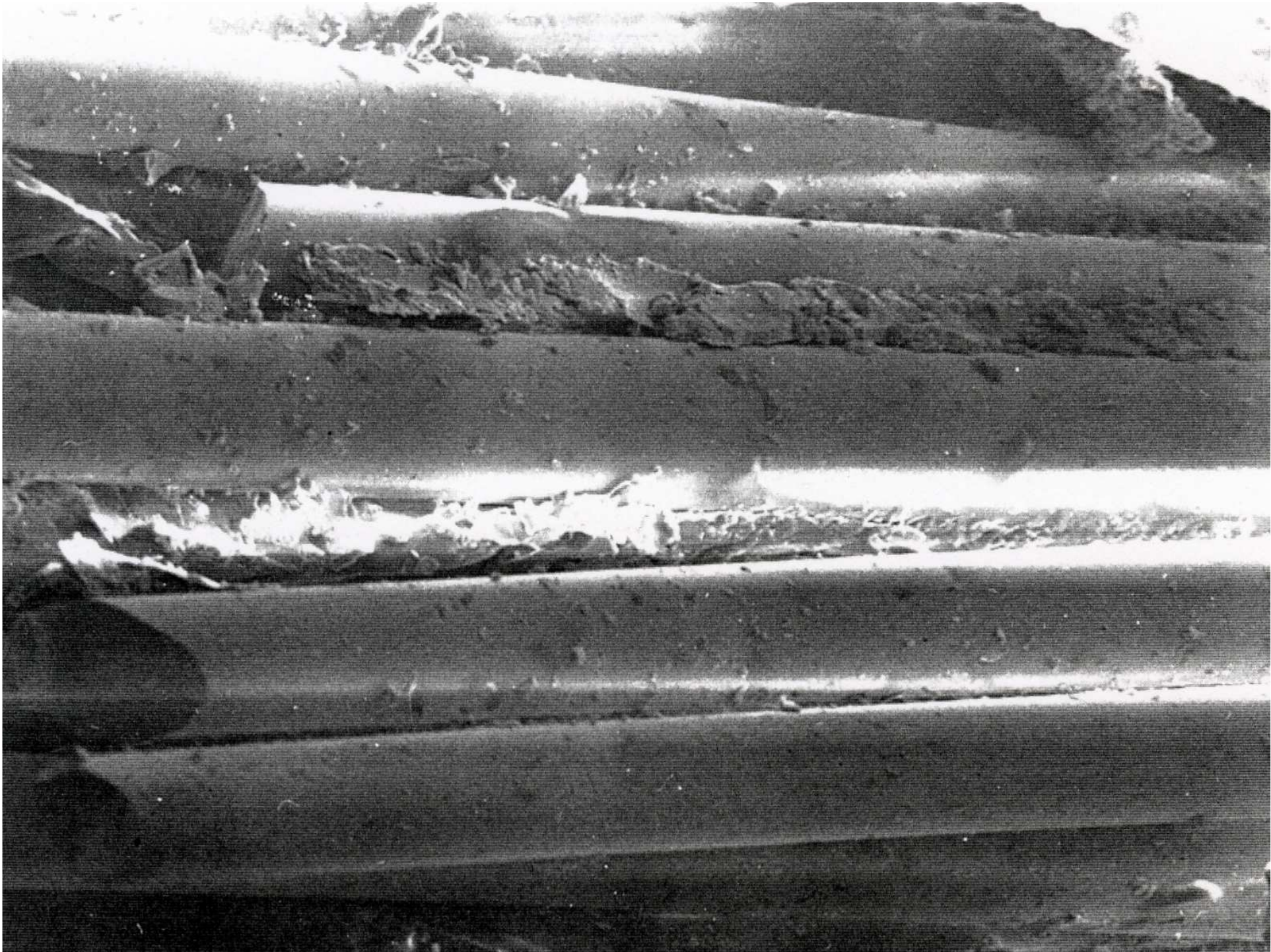


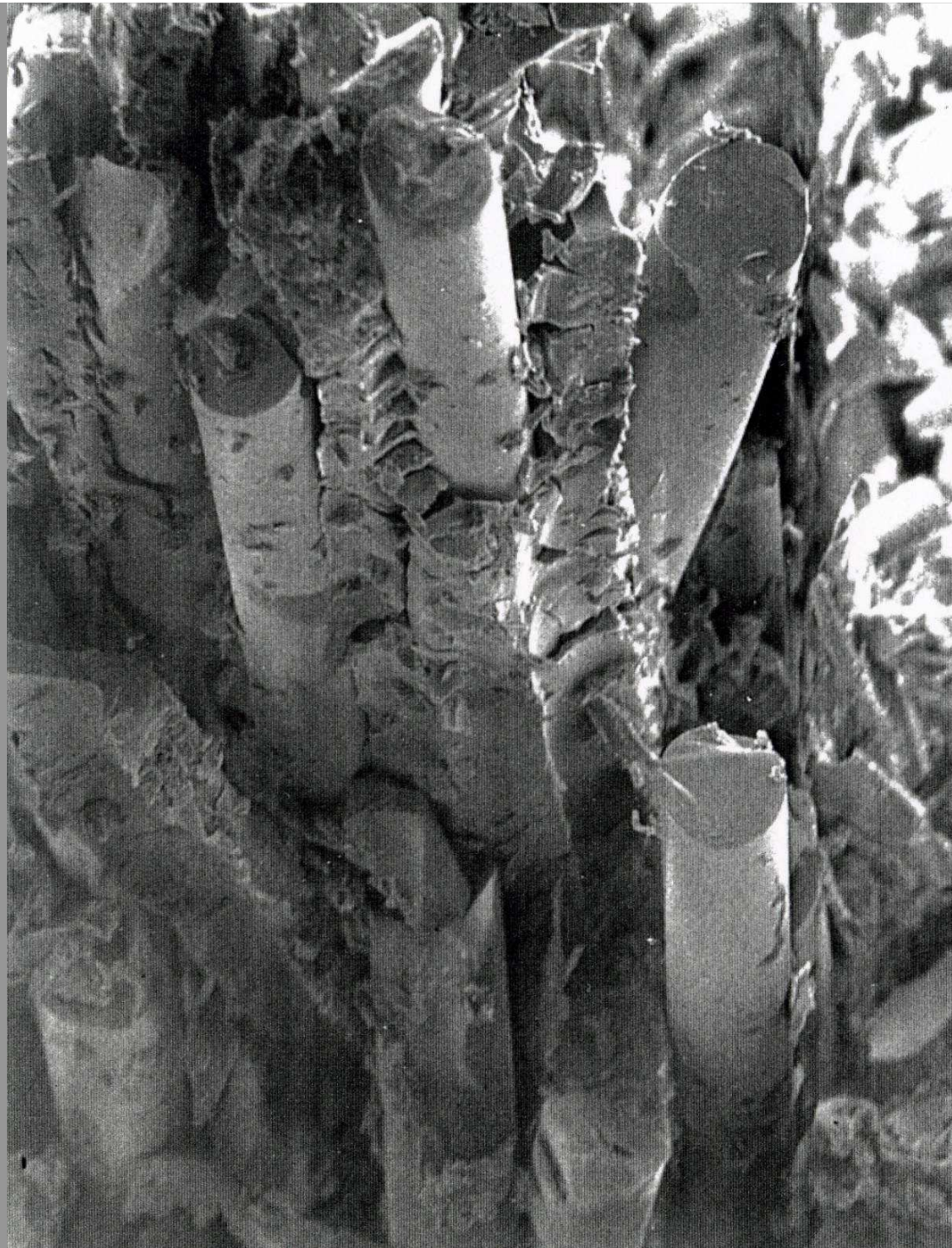


x2000
M20

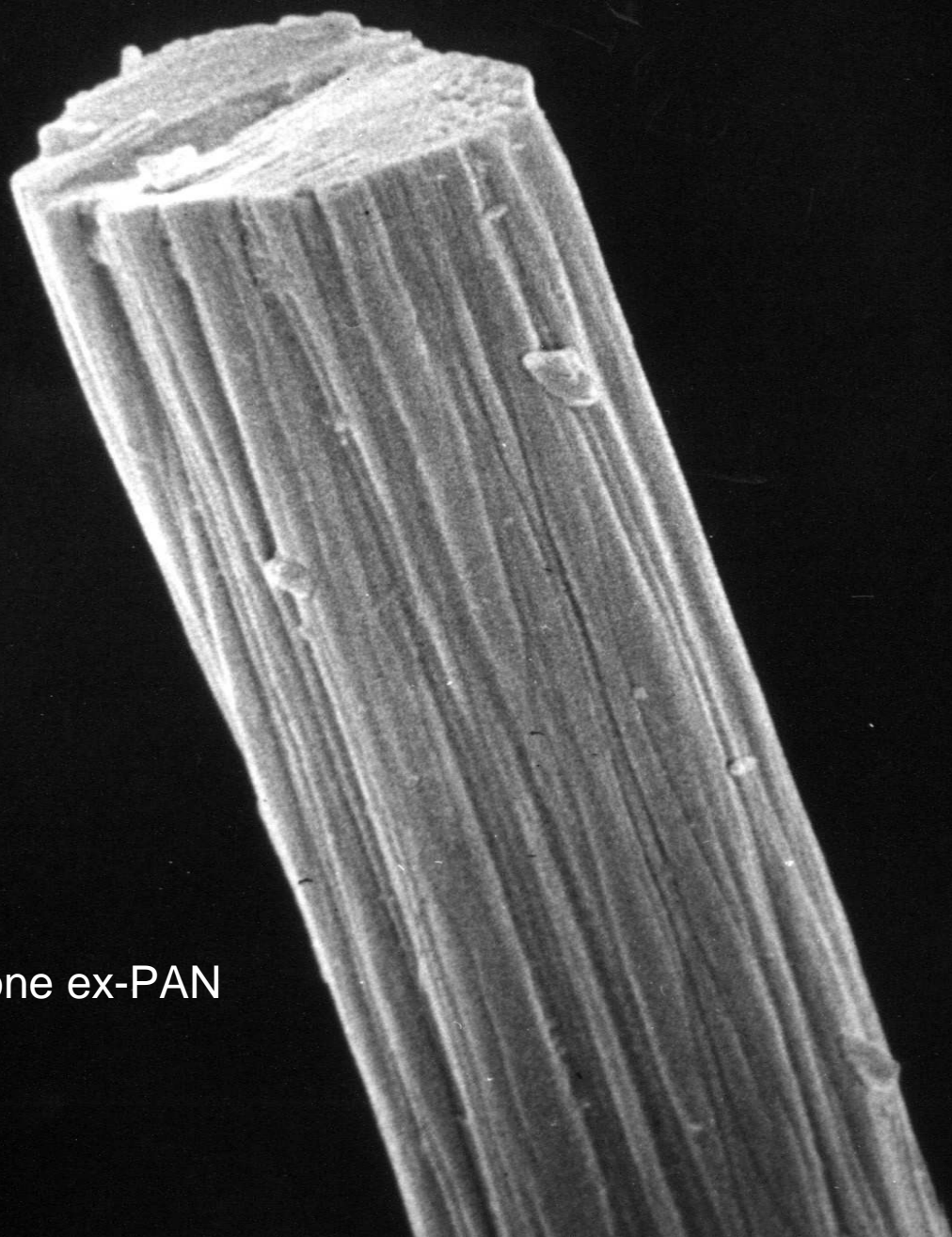
20 μm

4.00kV 13
MAP CDM ENSM





Rupture imprévue d'un composite à fibres de carbone



Fibre de carbone ex-PAN
 Φ 7 μ m

Cellulose

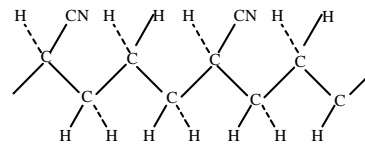


Regenerated cellulose

24% carbon

Carbon fibres with low mechanical properties but good thermal resistance

Oil



PAN

49% carbon

Carbon fibres with good mechanical properties but limited in high modulus range

Coal & steel industry

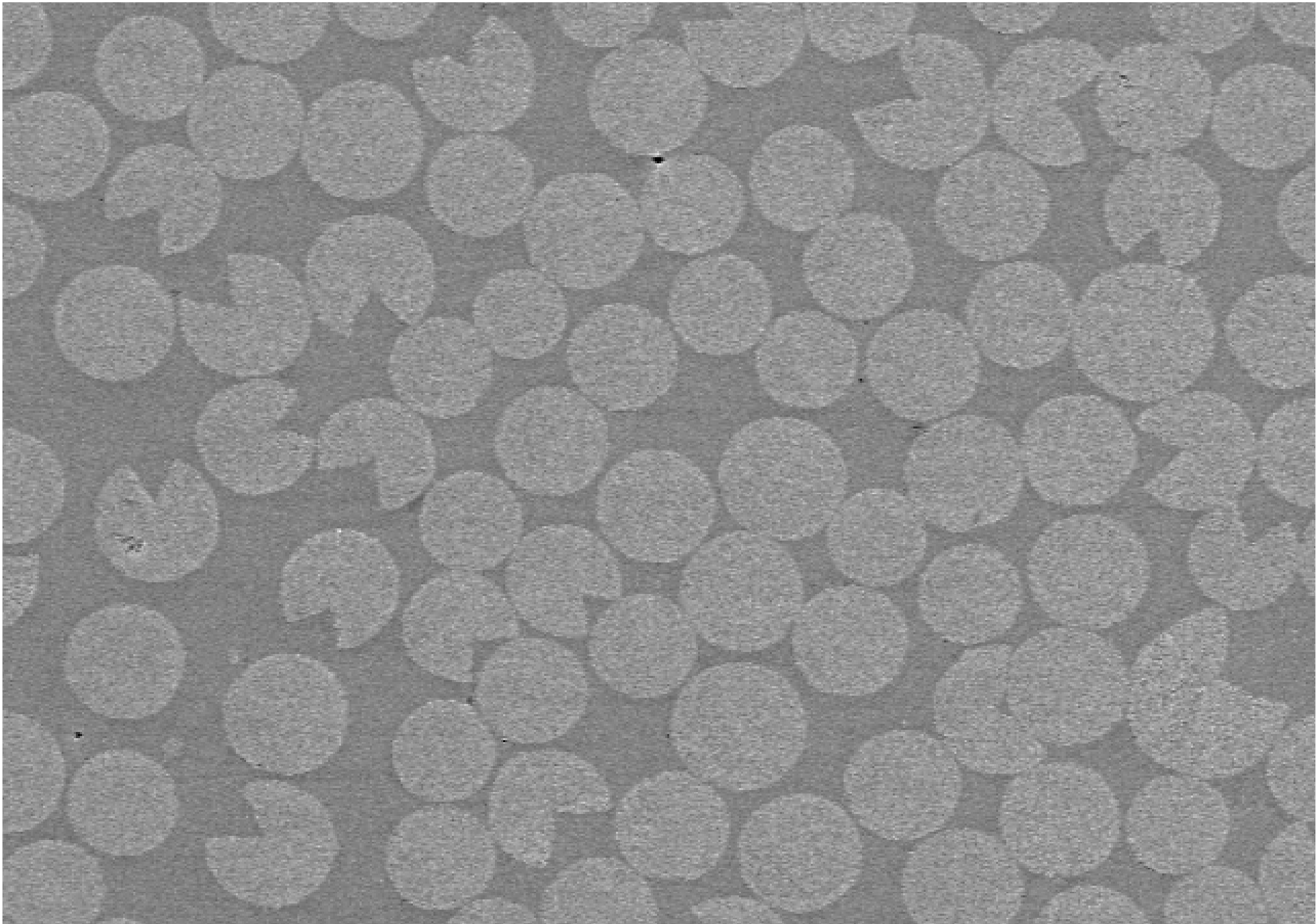


Oil refinement & coking process residues

Pitch

>80% carbon

Carbon fibres with very high moduli



x1000
#2

20 μ m
9 mm

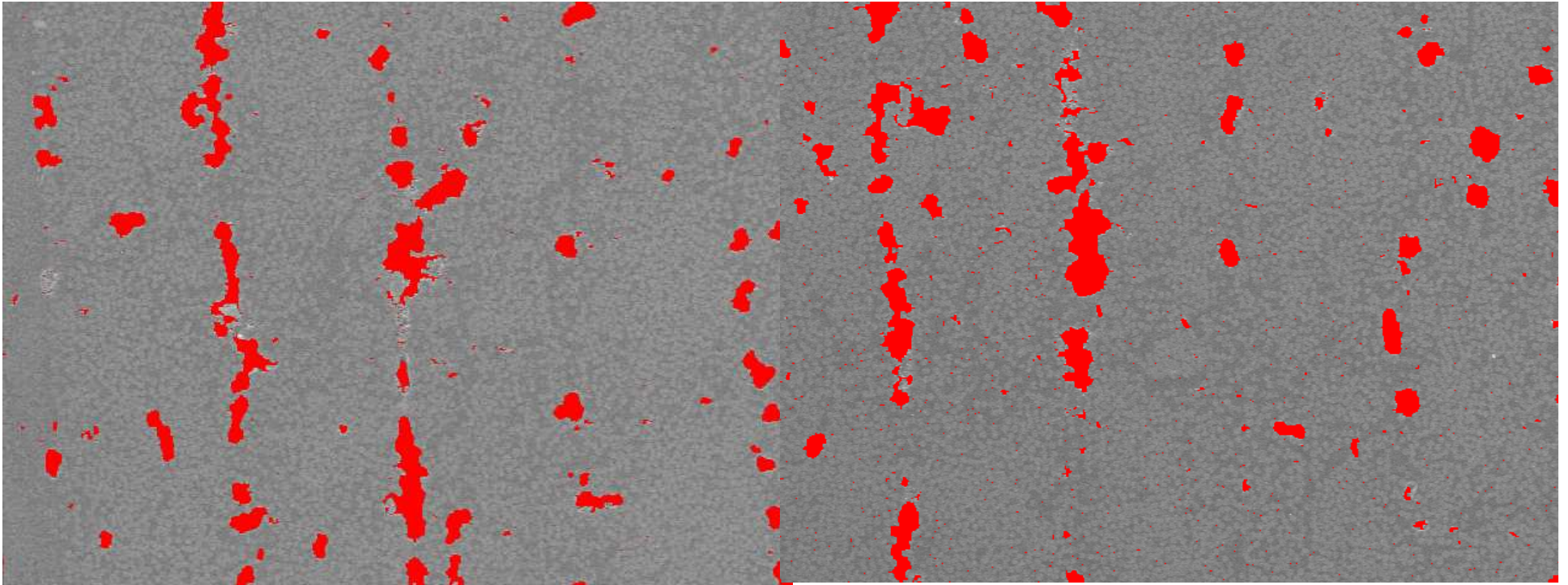
10kV
@CDM ENSMP MAP
8mm



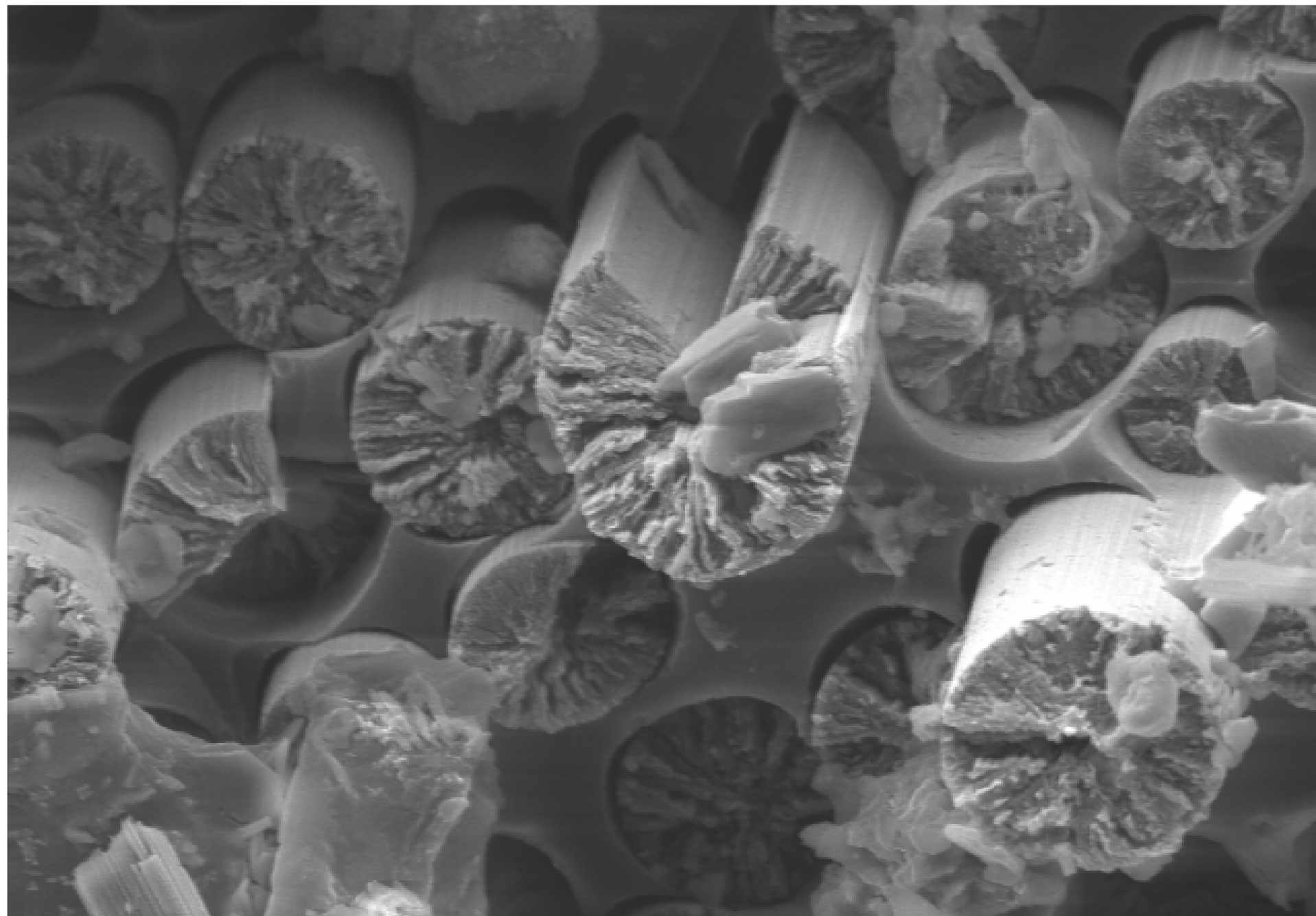
x2000
#2

20 μ m

10kV 16mm
@CDM ENSMP MAP



VARIATION DU TAUX DE POROSITE DE 4.7% à 5.8 %

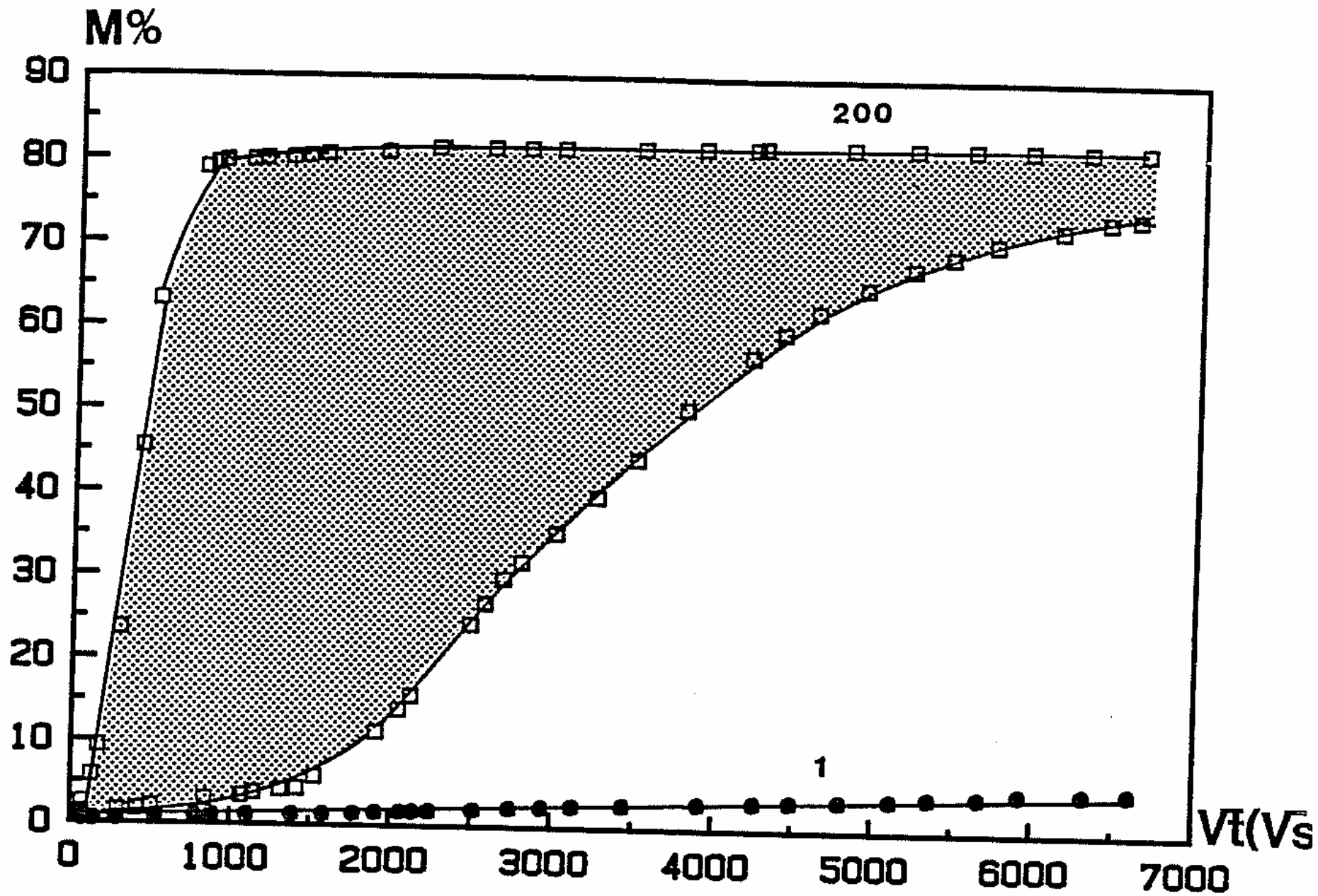


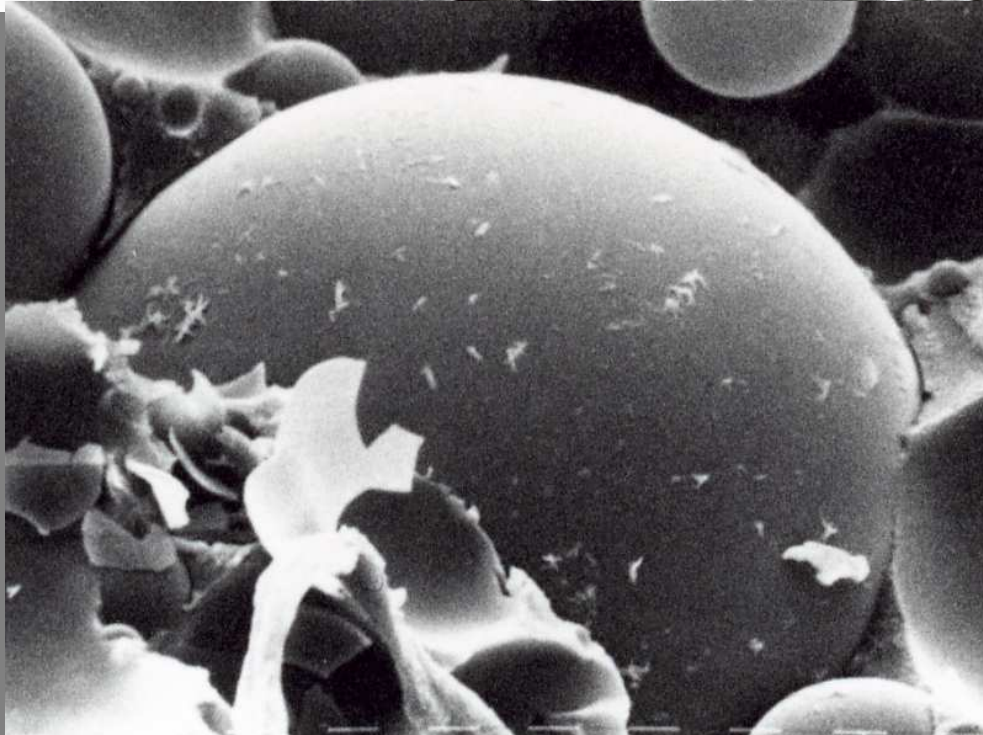
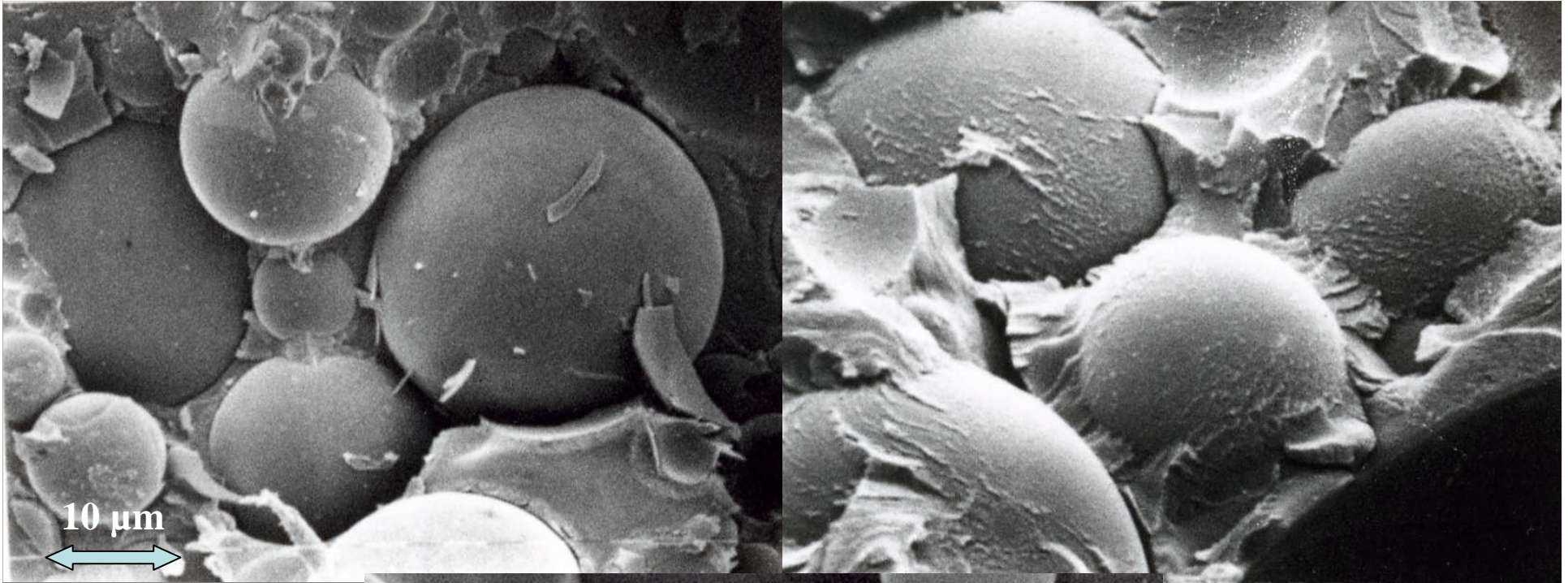
x2000
#2

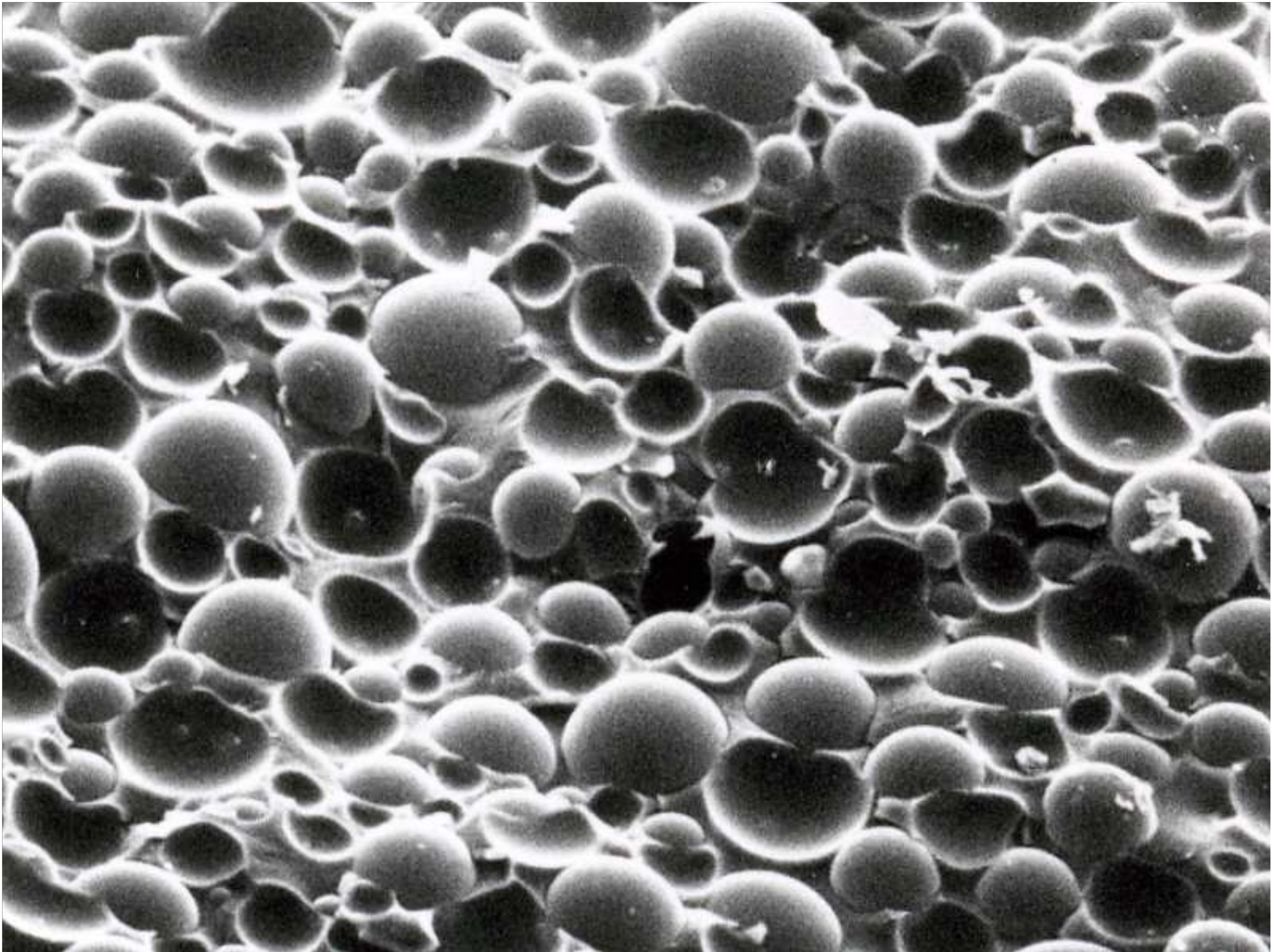
20 μ m

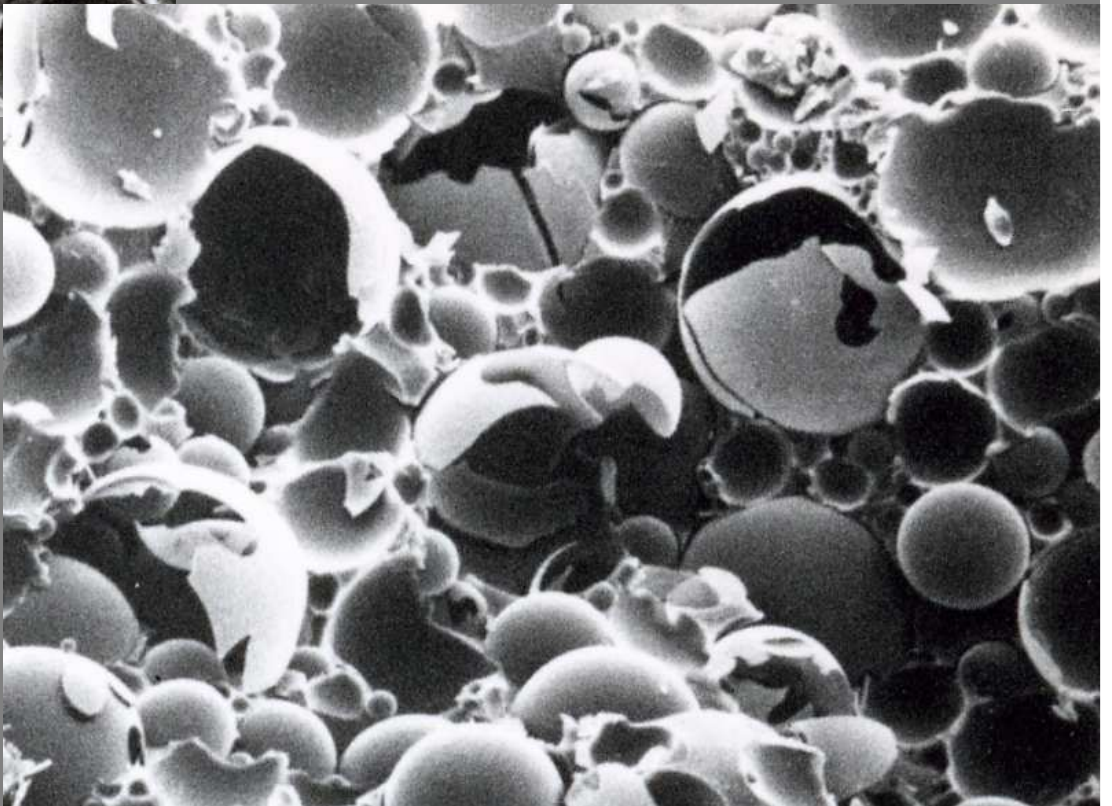
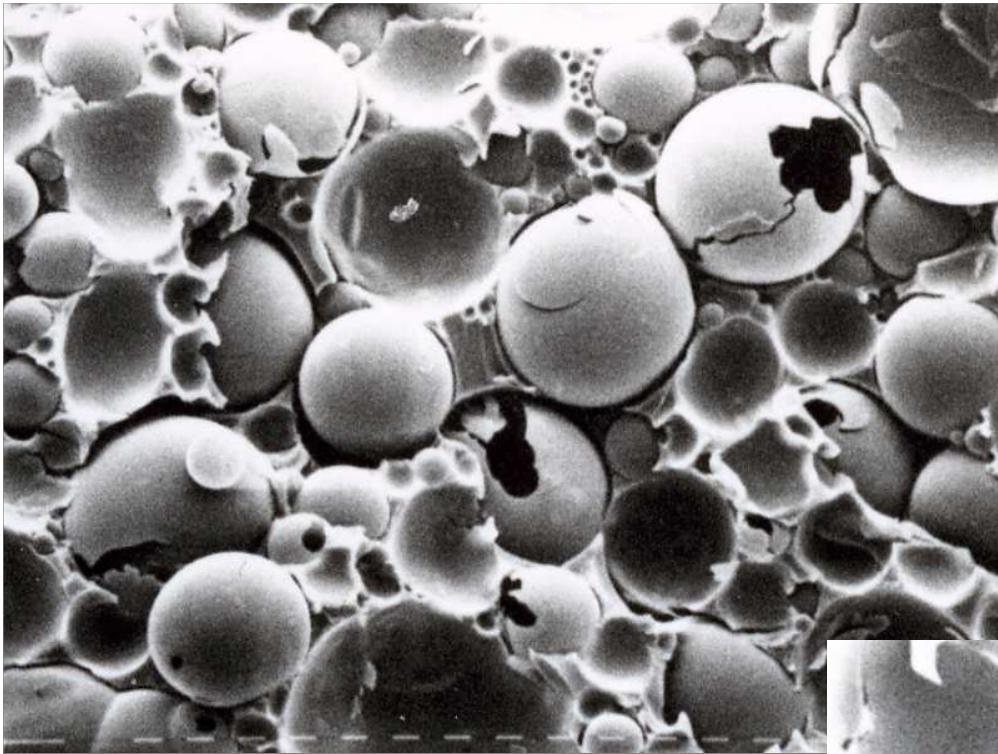
10kV 17mm
@CDM ENSMP MAP

La mousse syntactique

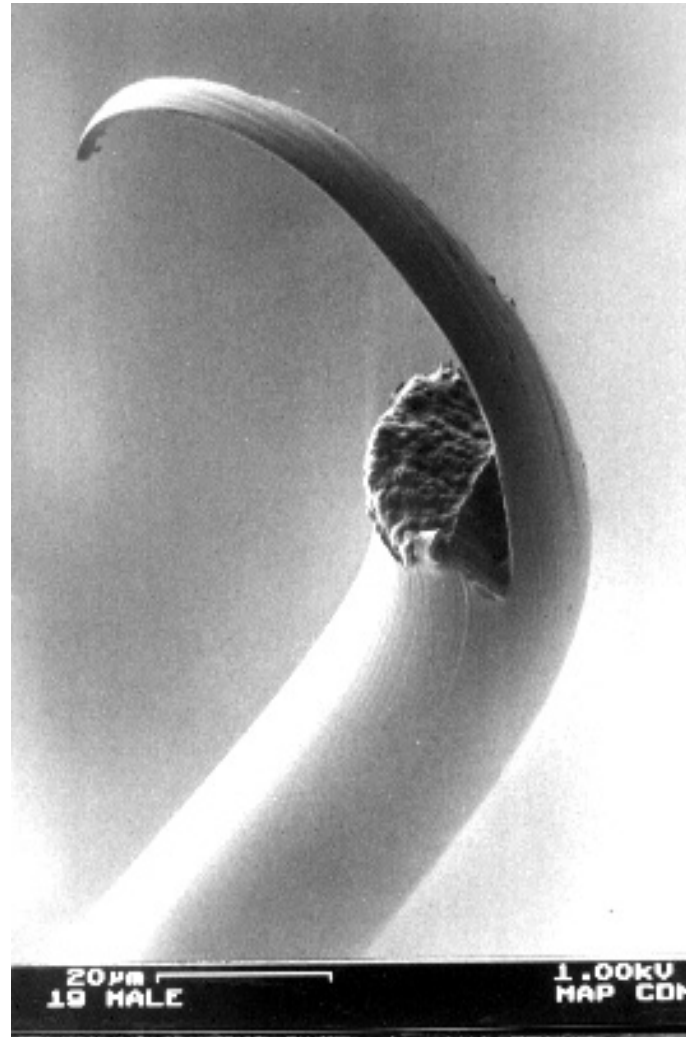
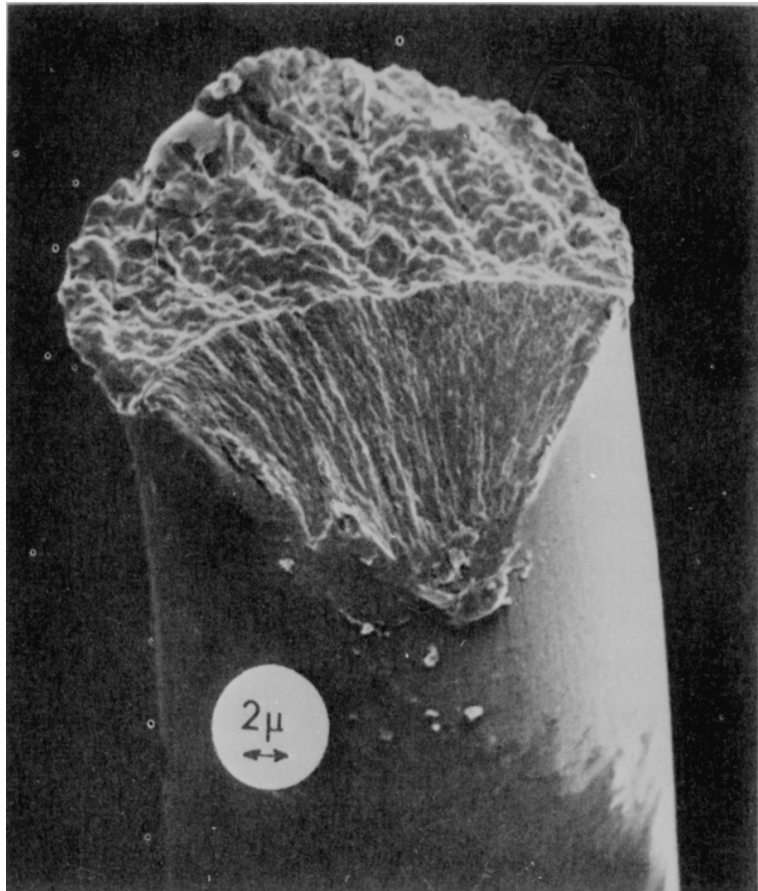


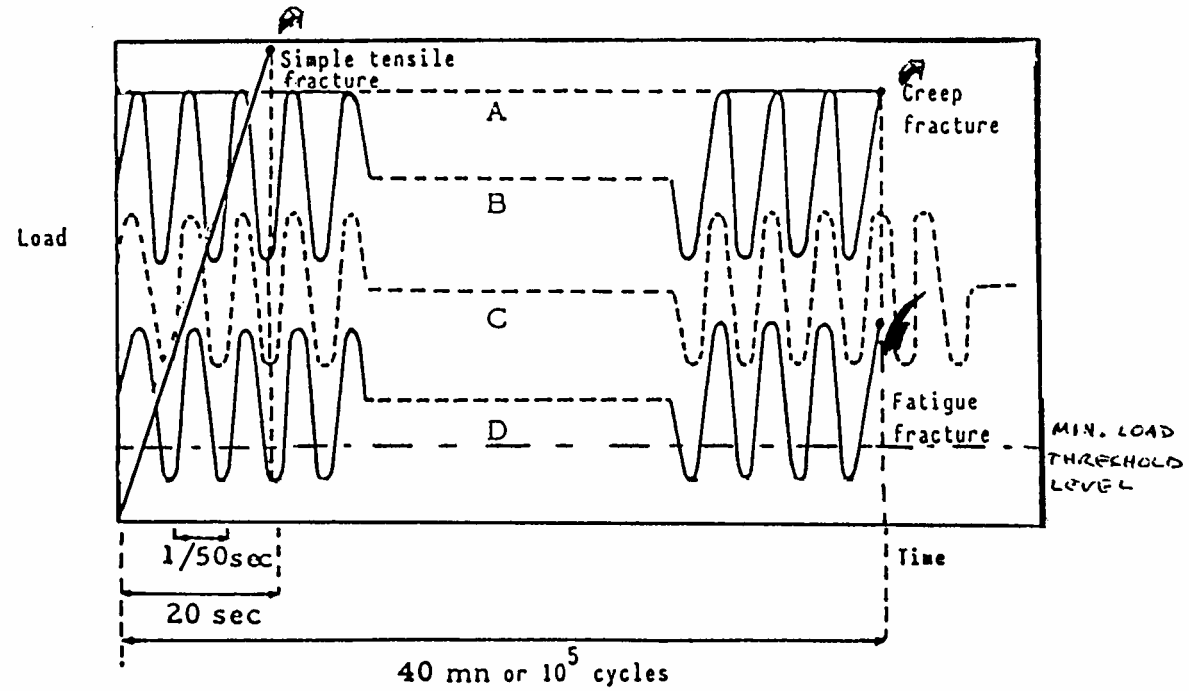




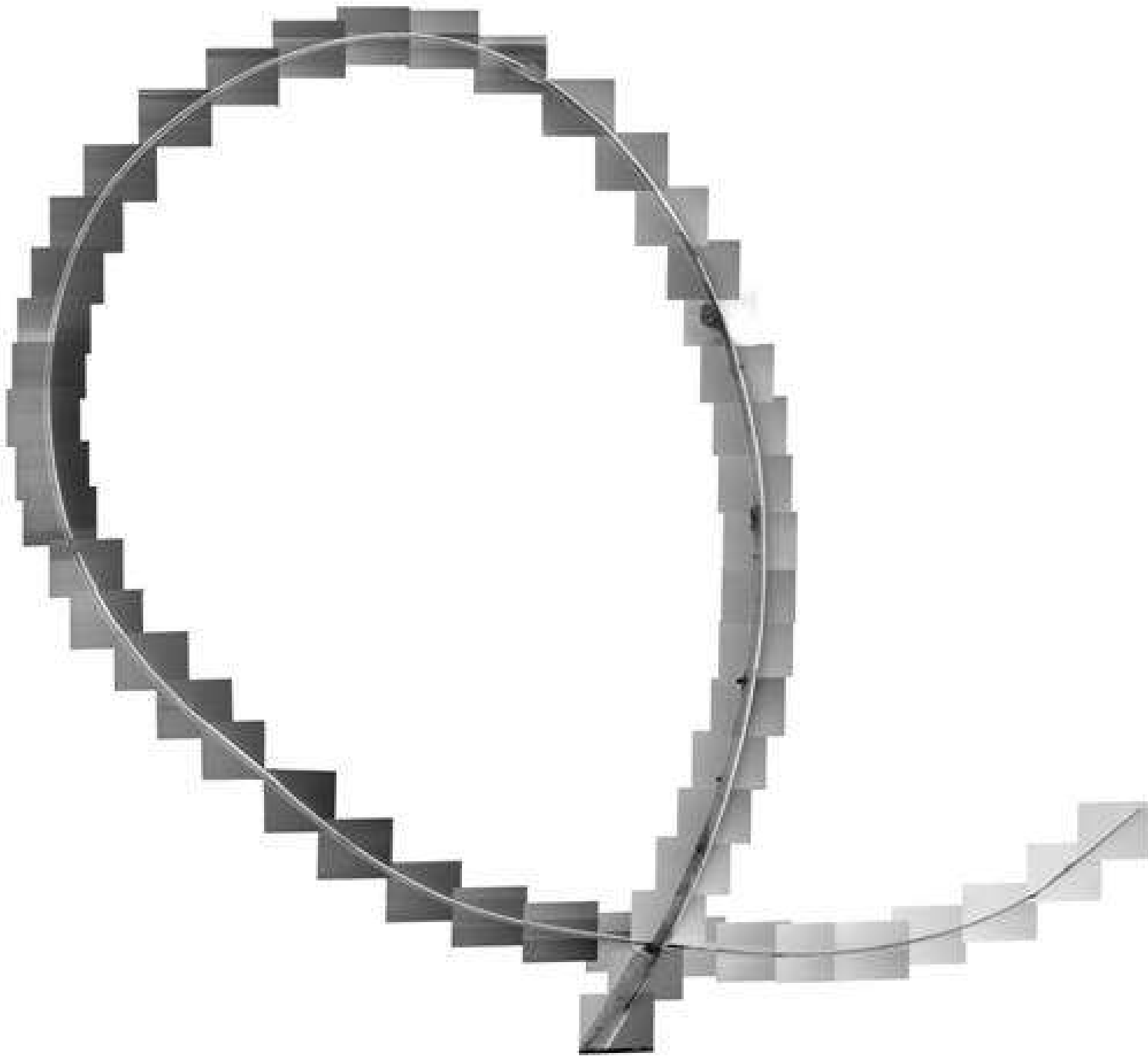


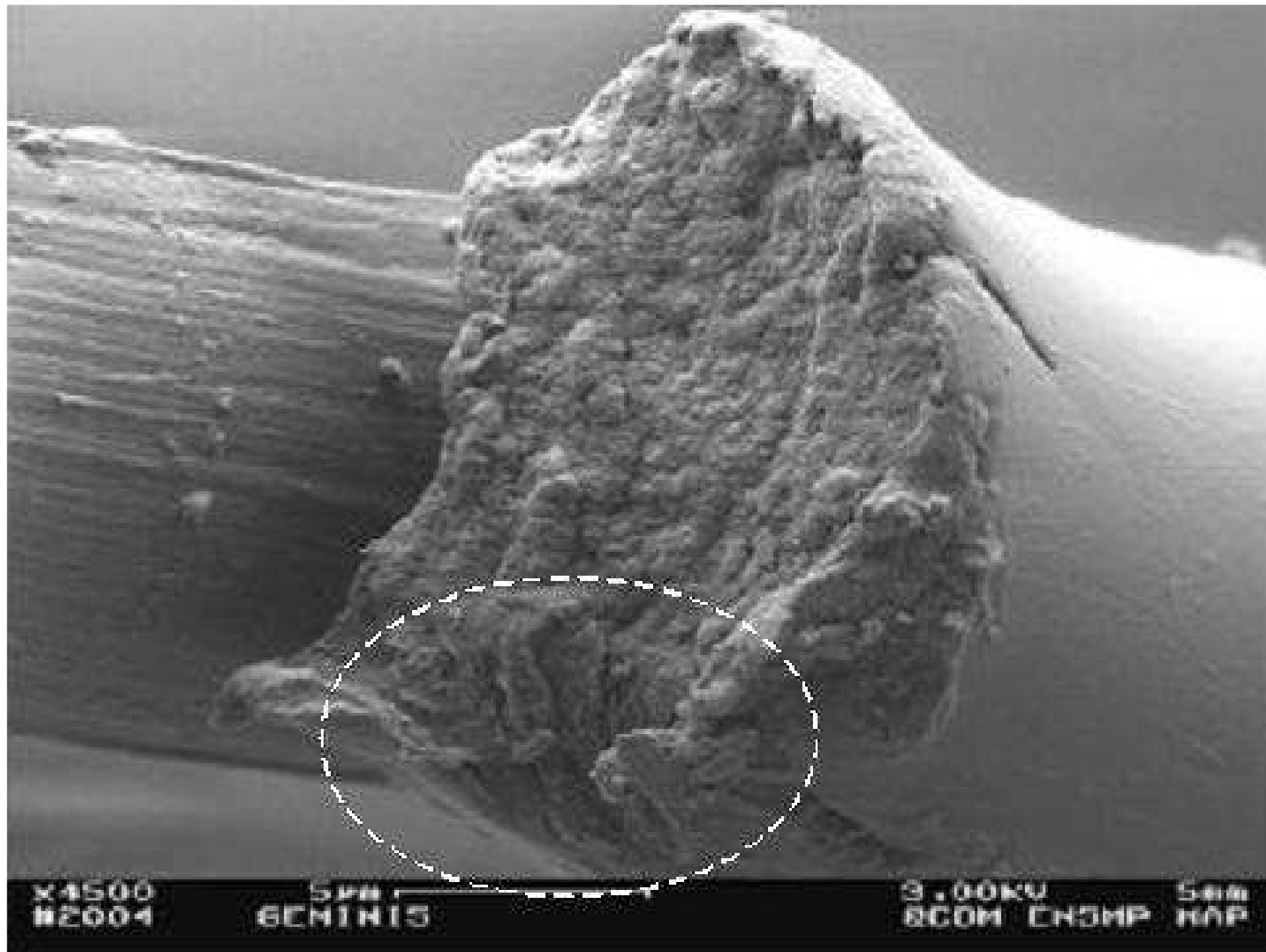
Rupture des fibres PET et PA noyées dans du caoutchouc

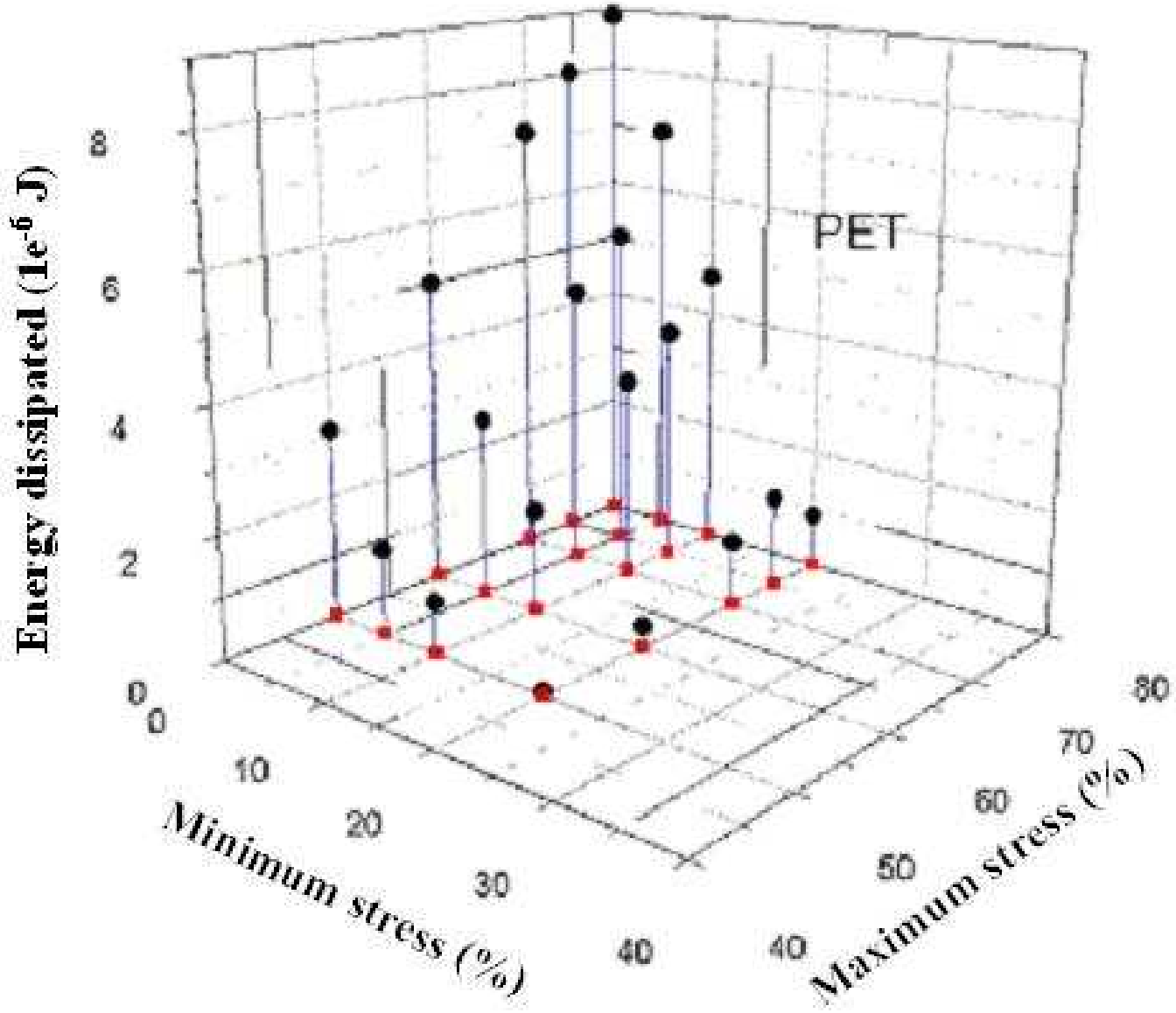


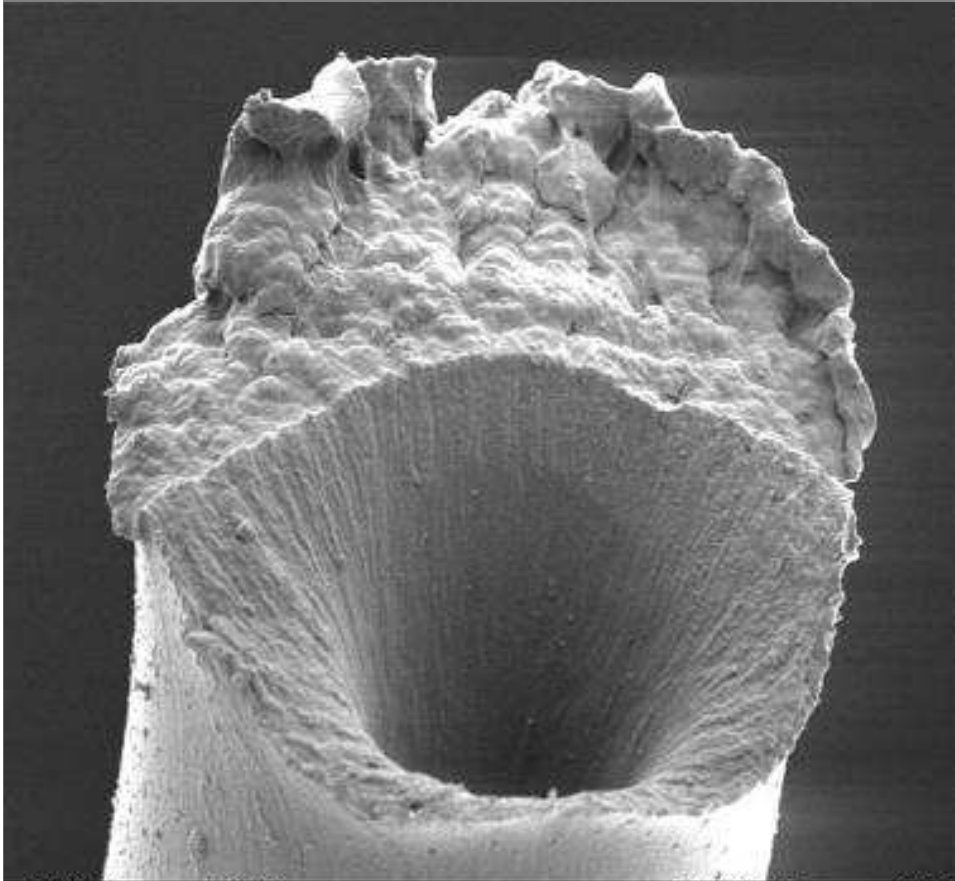


Schematic diagram of the effects of tensile, creep and cyclic loading at 50.Hz on nylon 66 fibers.



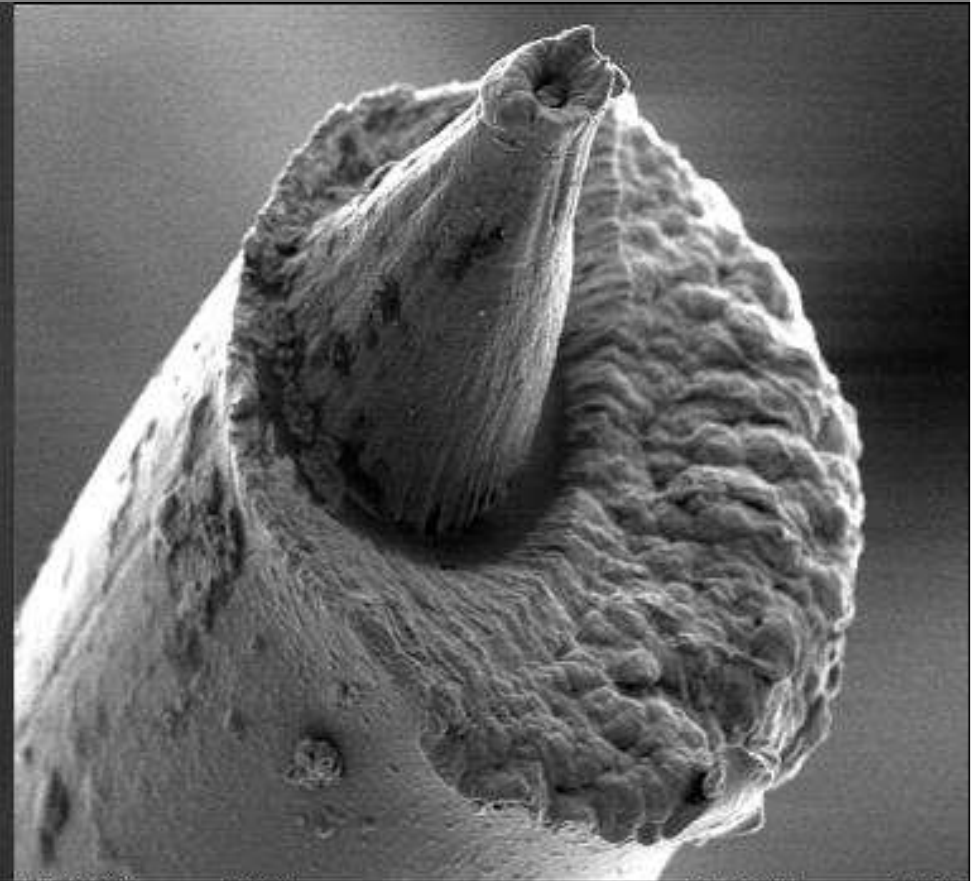






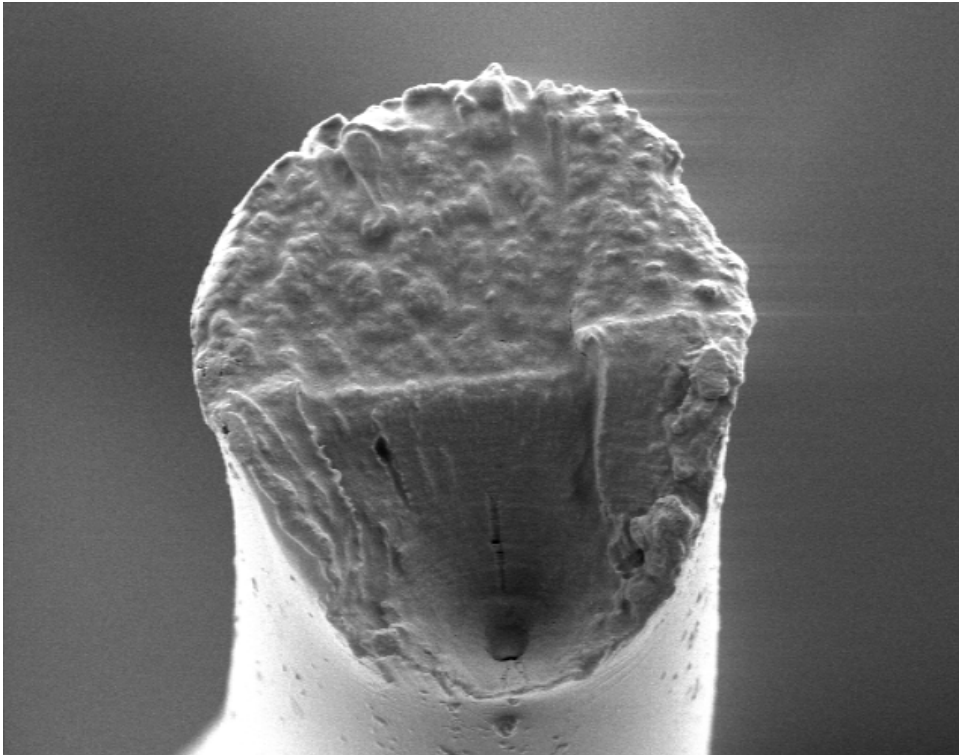
x3000
#2
10µm
NYL33 (0-72%)

3.00kV 7mm
@CDM ENSMP MAP

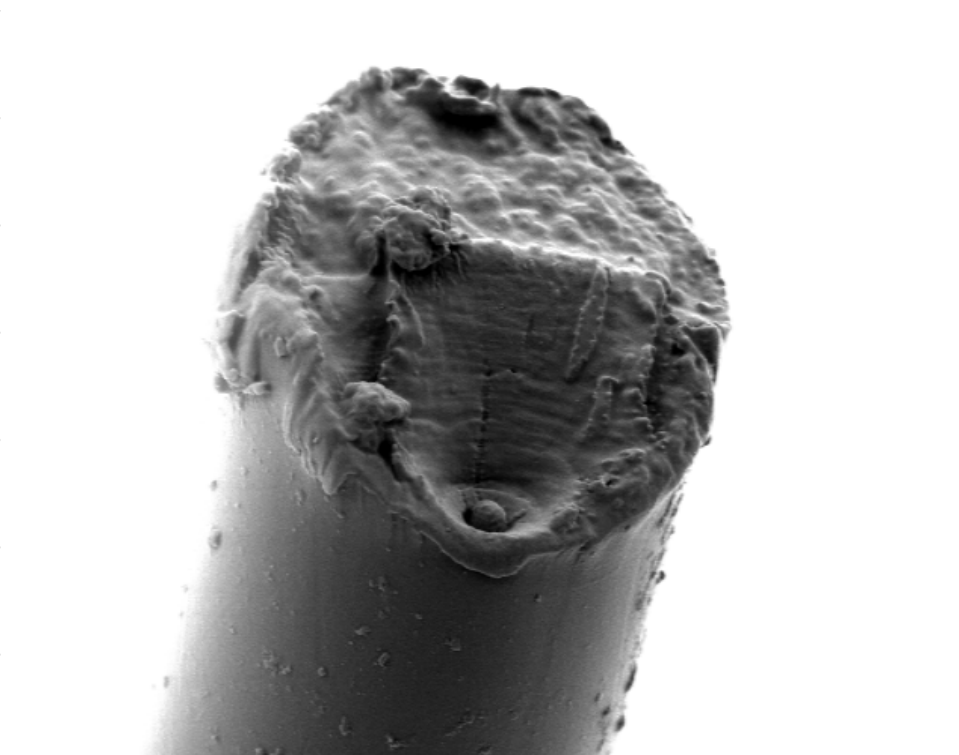


x3000
#2
10µm
NYL33 (0-72%)

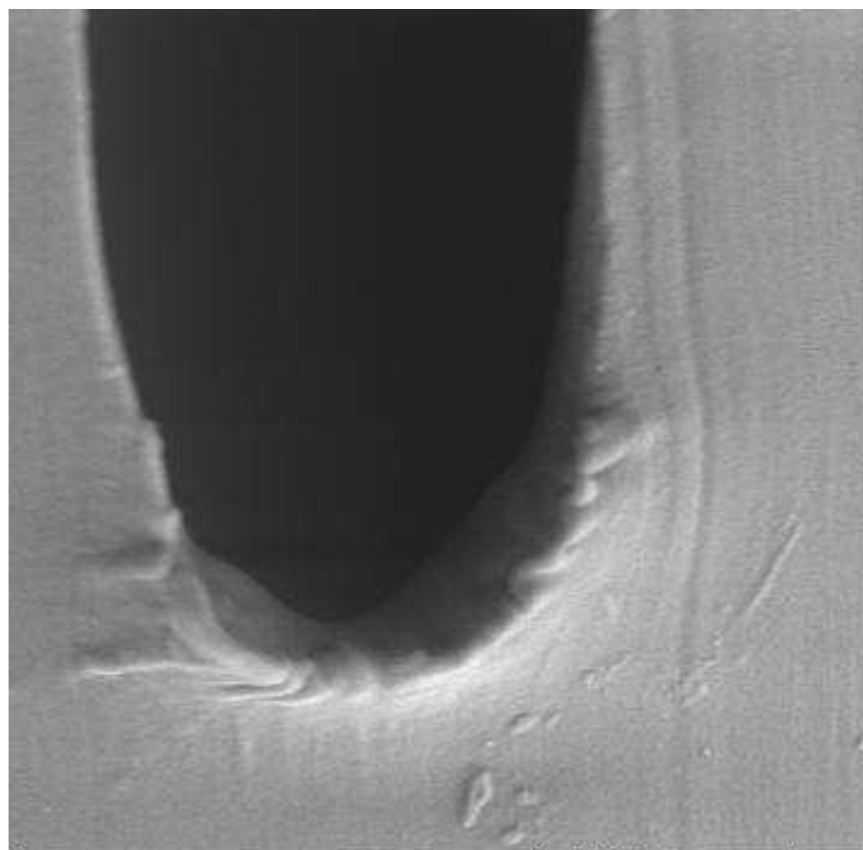
3.00kV 6mm
@CDM ENSMP MAP



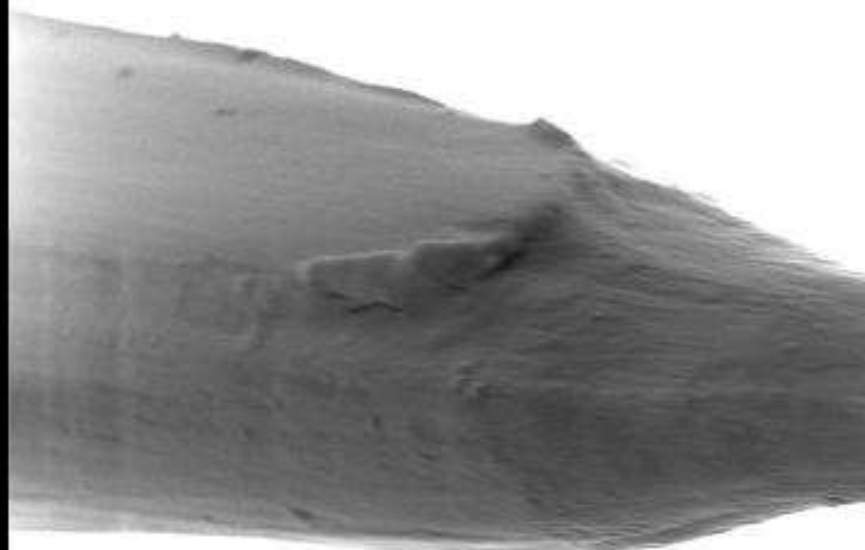
x3000 10µm 3.00kV 4mm
#2 PEI73 <0-85%> @CDM ENSMP MAP



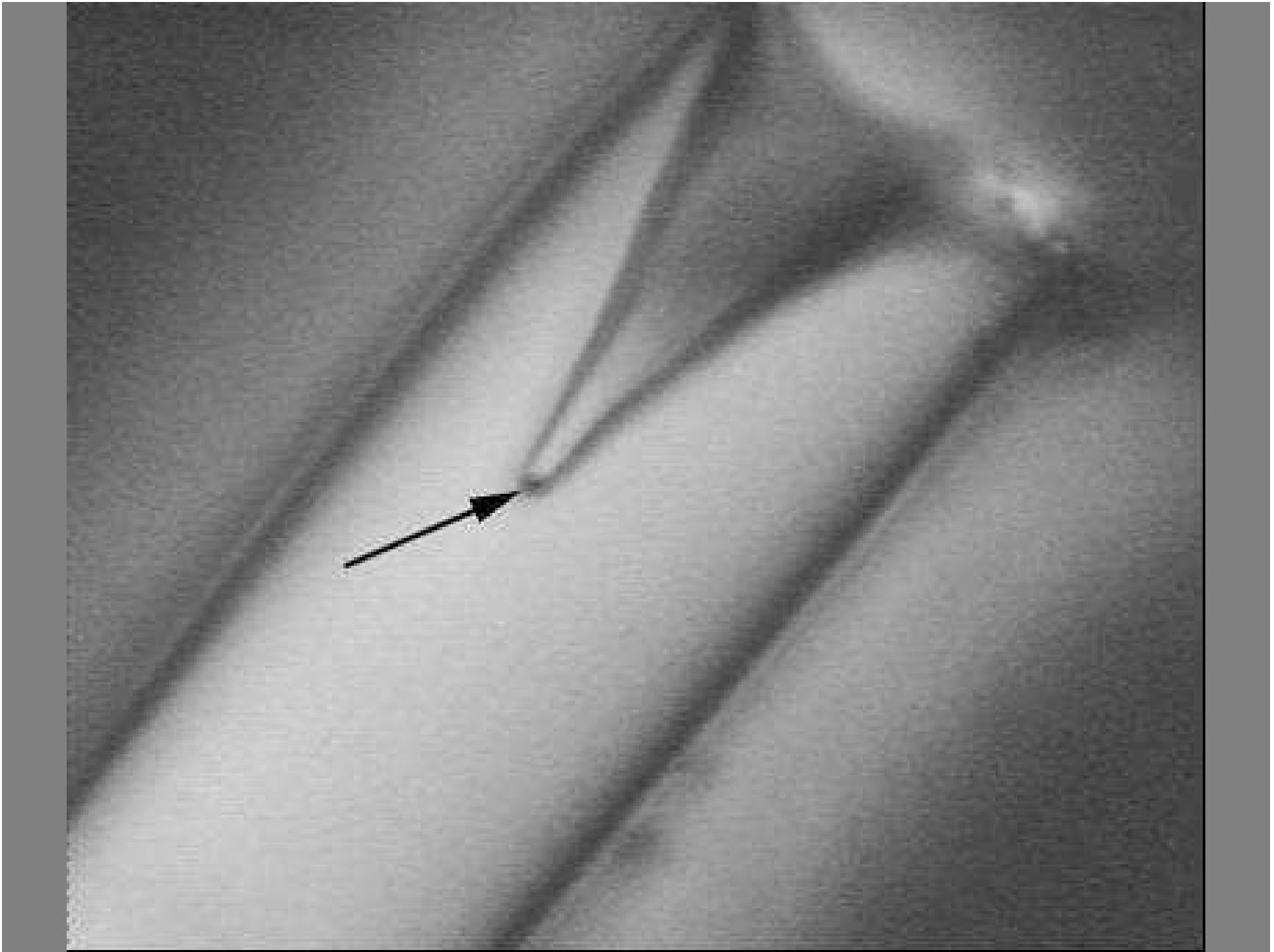
x3000 10µm 3.00kV 4mm
#2 PEI73 <0-85%> @CDM ENSMP MAP



2 μm | 3.00kV 4mm
NYL131 (0-80%) @CDM ENSMP MAP

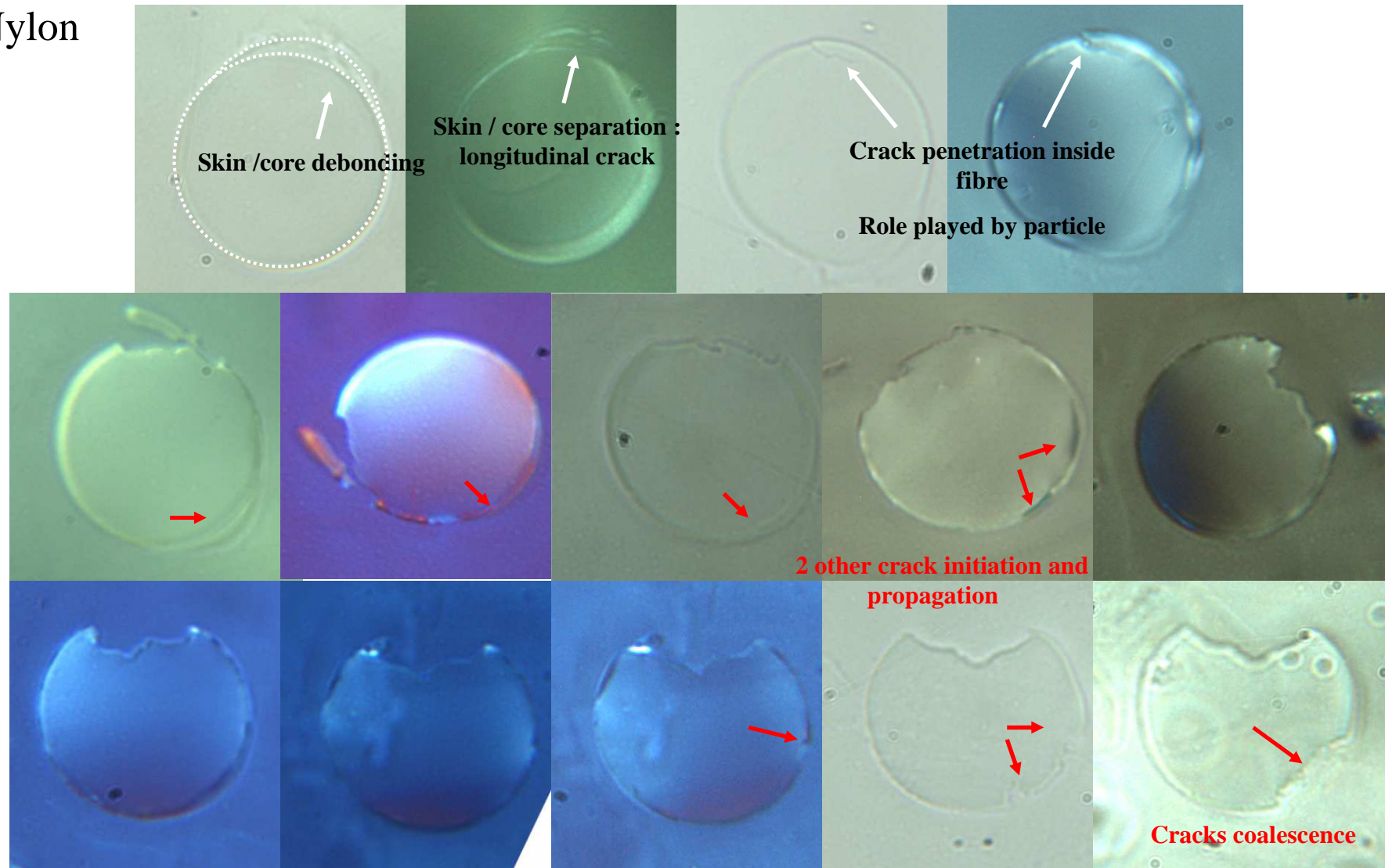


x10000 2 μm | 3.0
#1 NYL131 (0-80%) @CD



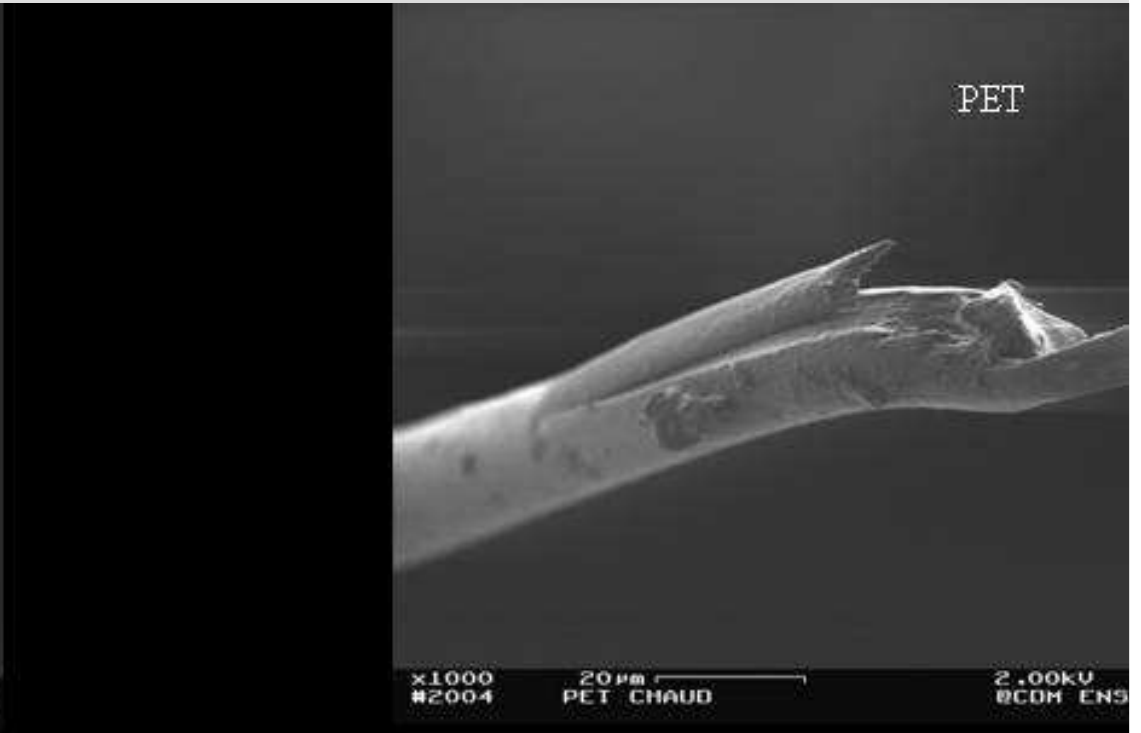
Room temperature mechanical fatigue : microtomy sections

Nylon



- skin /core structure existence with weak interface

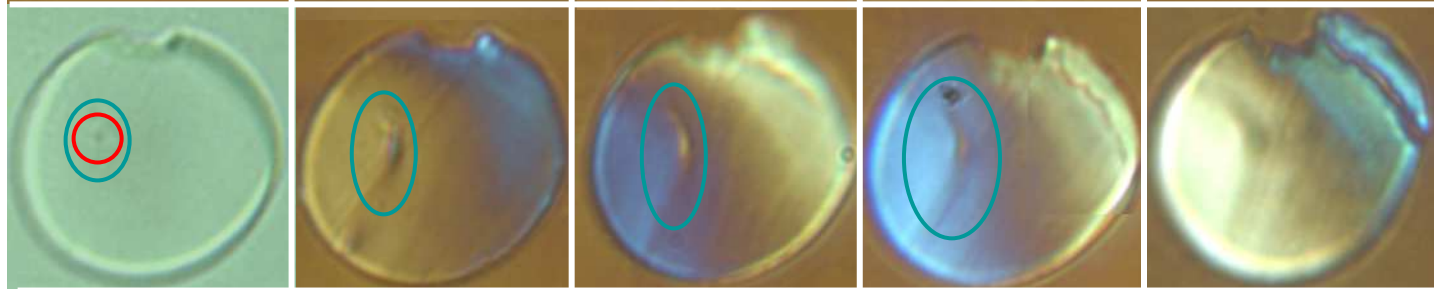
- particle important role for fatigue crack propagation



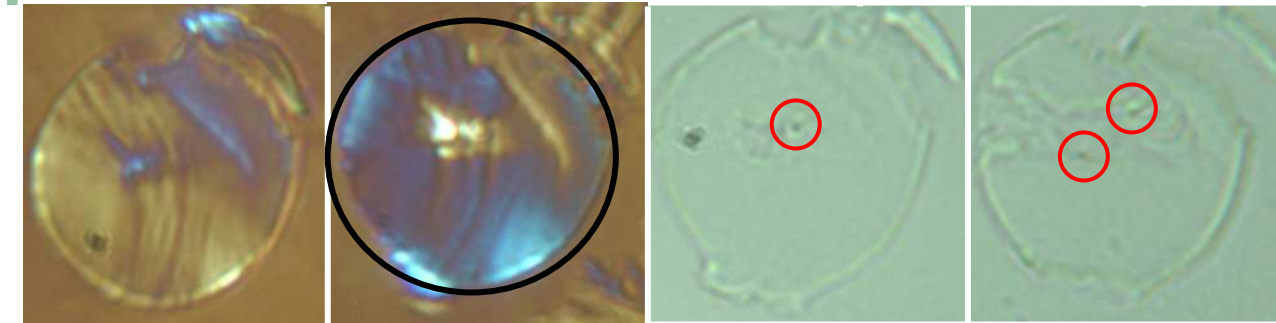
Mechanical fatigue in hot conditions (PET 120°C) : microtomy sections



①) Classical crack as at room temperature

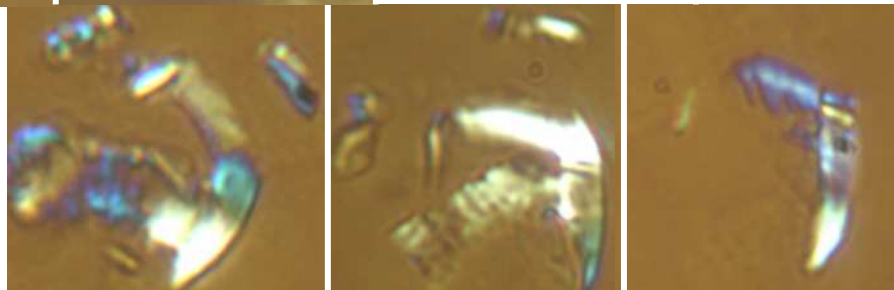


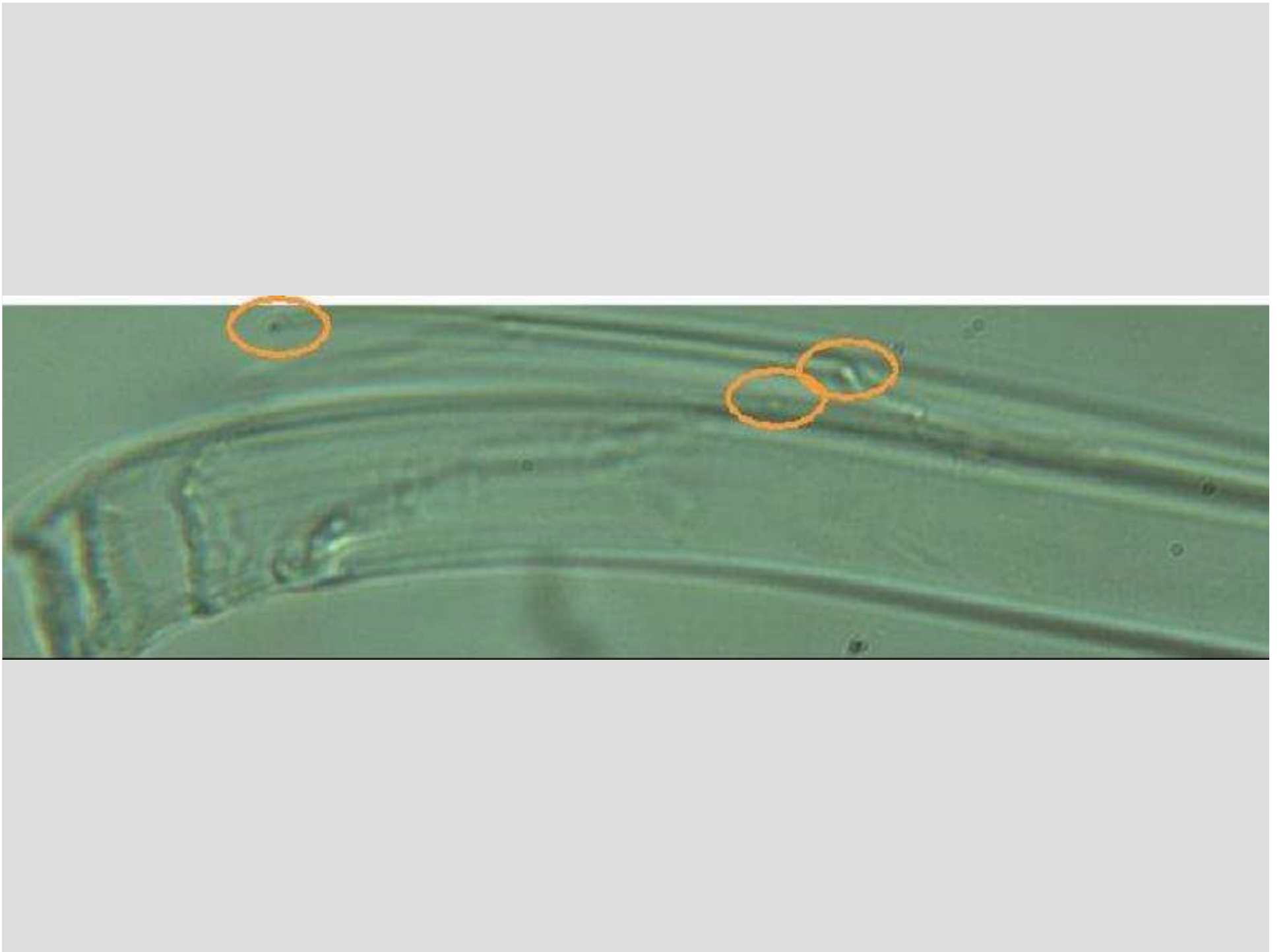
②) Internal longitudinal crack



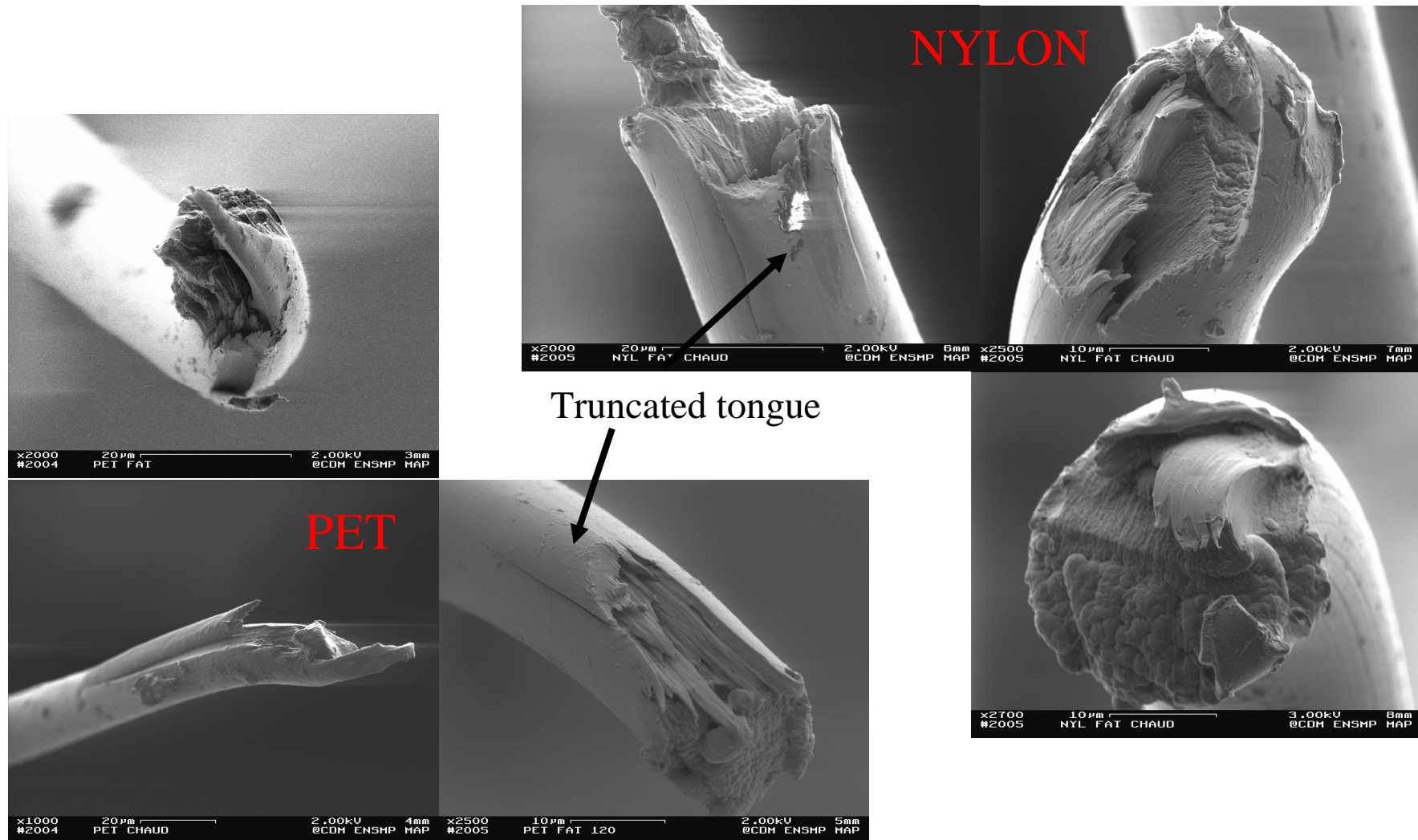
③) Role played by solid inclusions

④) Whole fibre affected





Mechanical fatigue in hot conditions (80°C)



2 symmetrical fracture morphologies with rather thick truncated tongues, multi longitudinal cracks around mean crack and accelerated creep phenomena

Rupture des fibres céramiques

Production of the first ceramic reinforcements for CMC
"SiC" fibres - Pr YAJIMA (Tohoku University) 1976

Nippon Carbon 1982

Spinning SiC Precursor:

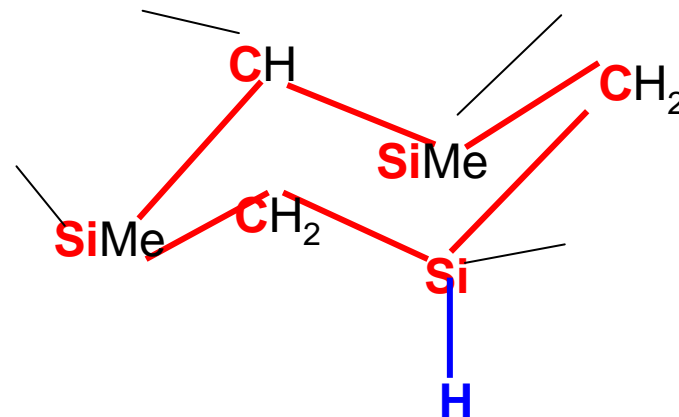
PolyCarboSilane

Fusible
precursor fibre

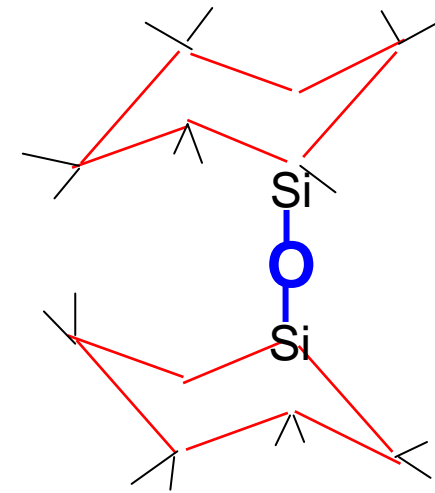
Cross-linking
by oxidation

Infusible
during heat treatment

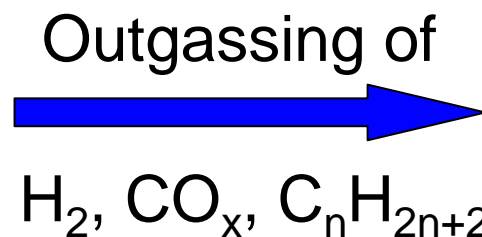
Heat treatment
up to 1200°C



PCS
Mw ≈ 1500

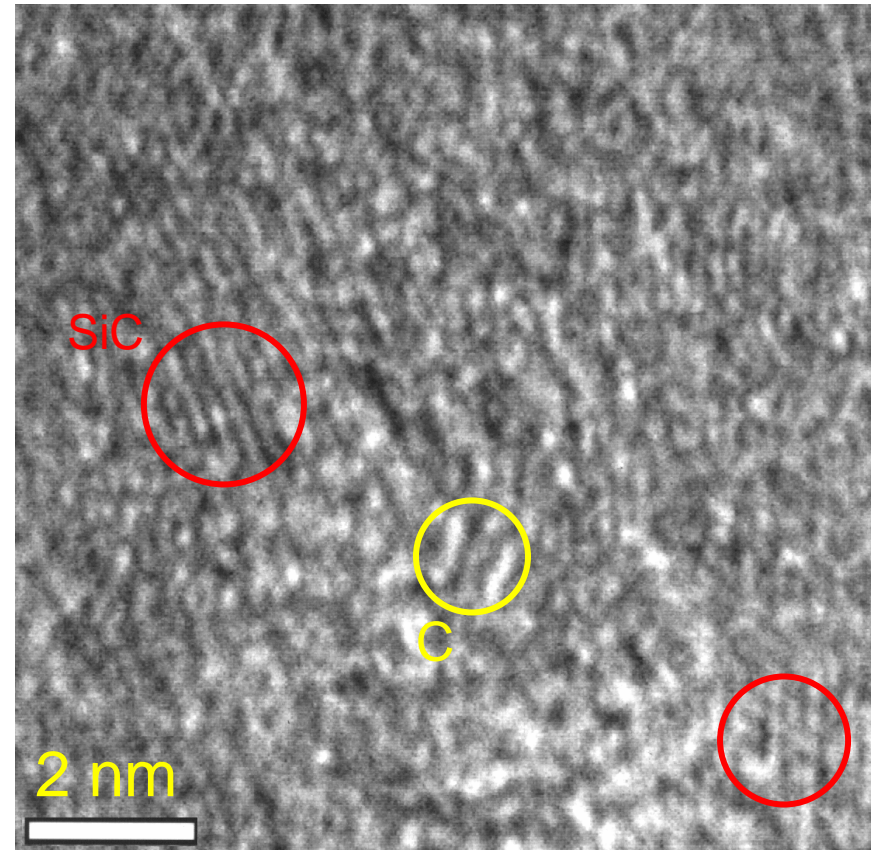
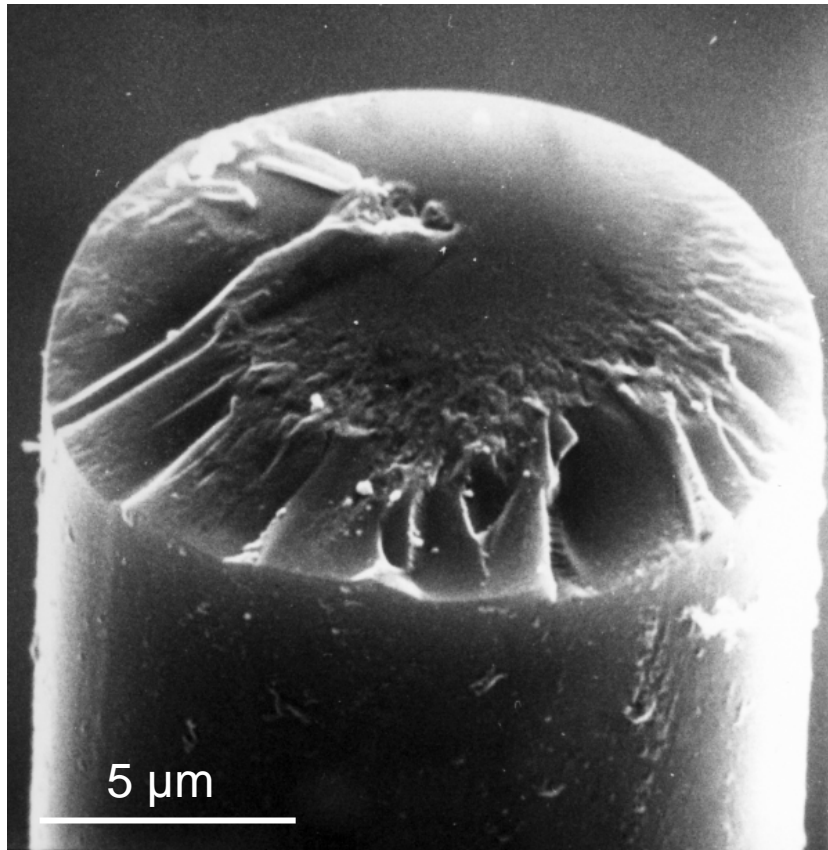


Cross-linking
200°C in air



Nicalon fibre
SiC fibre ?

Nicalon NL 200 fibre



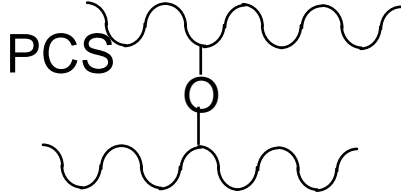

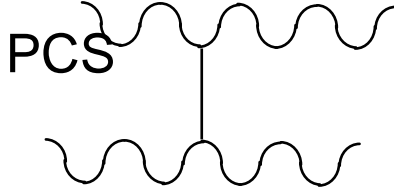
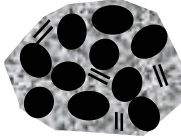
$D = 15 \mu\text{m}$, $\rho = 2.55 \text{ g/cm}^3$
 $E = 180 \text{ GPa}$, $\sigma = 2 \text{ GPa}$

$\text{SiC} < 2 \text{ nm}$, $\text{C} < 1 \text{ nm}$,
+Si-C-O metastable intergranular phase

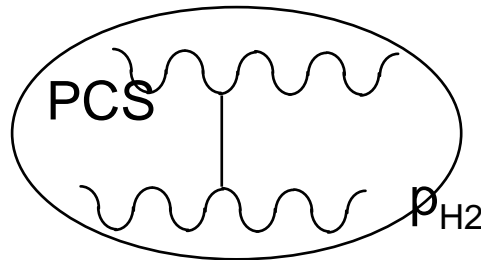
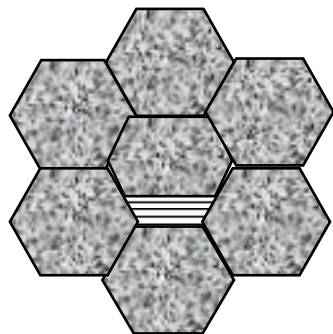
900°C : Creep

1500°C: Degradation, uncontrolled grain growth

Trois Générations de fibres SiC

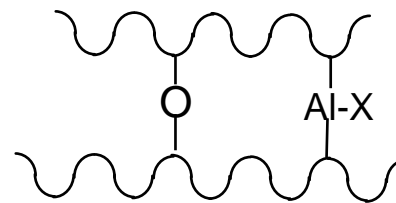
 <p>PCS</p> <div style="border: 1px solid black; padding: 2px; display: inline-block;">NL-200</div>	 <p>SiC 2nm + C + Si-C-O</p> <p>12%wt O, C/Si=1.31 E≈200 GPa</p> <p style="text-align: center;">Première génération Réticulation par oxygène</p>
<p style="text-align: center;">Deuxième génération Réticulation par bombardement électronique</p>	 <p>PCS</p> <div style="border: 1px solid black; padding: 2px; display: inline-block;">Hi-Nicalon</div>  <p>SiC 5-20nm + C + Si-C</p> <p>0.5%wt O, C/Si=1.39 E≈260 GPa</p>

SiC 50-200nm : E≈340 – 390 GPa

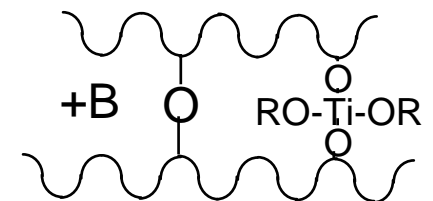


Hi-Nicalon S

Troisième génération

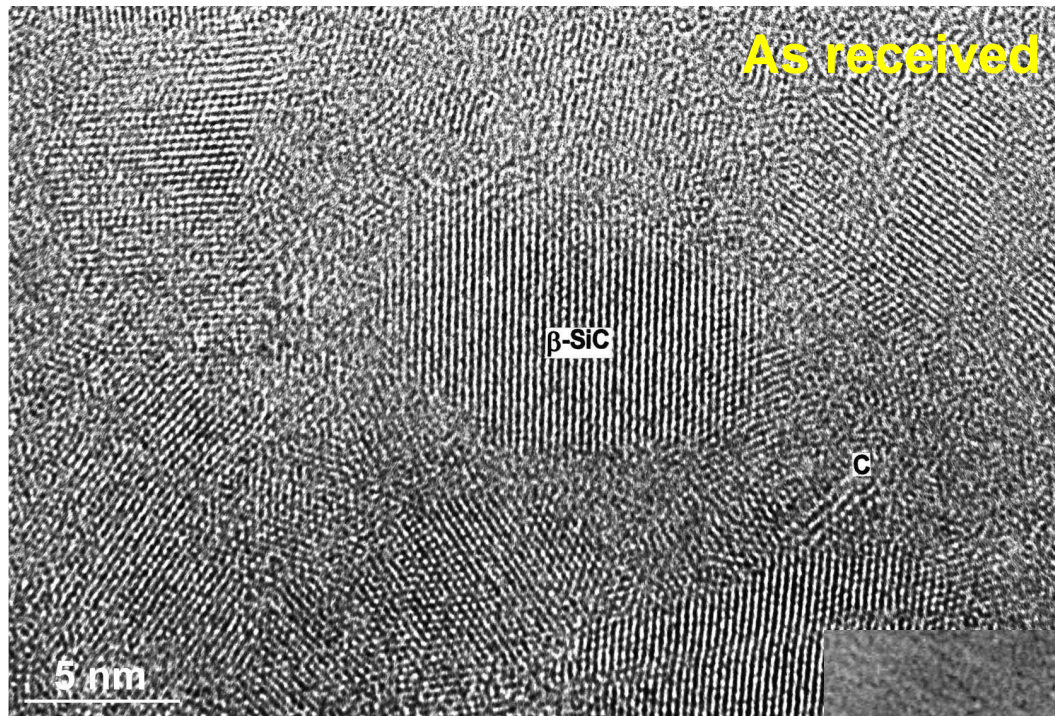


Tyranno-SA



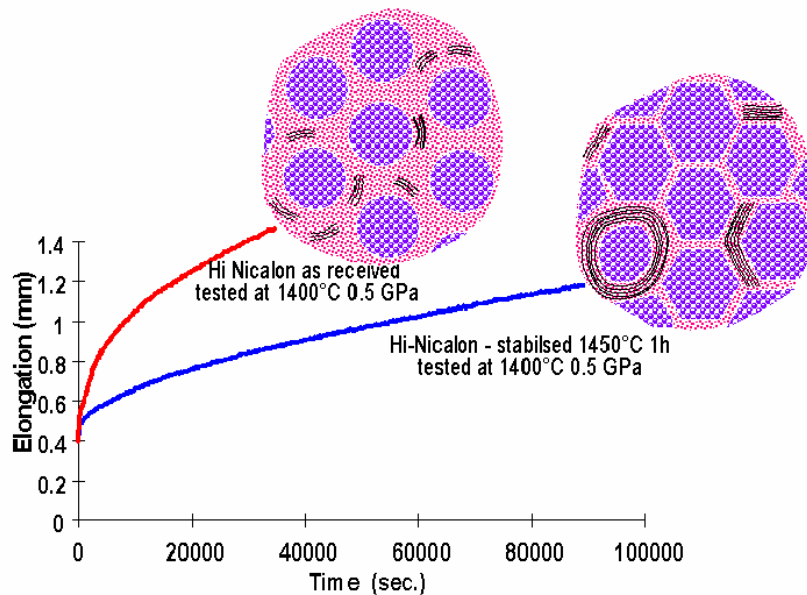
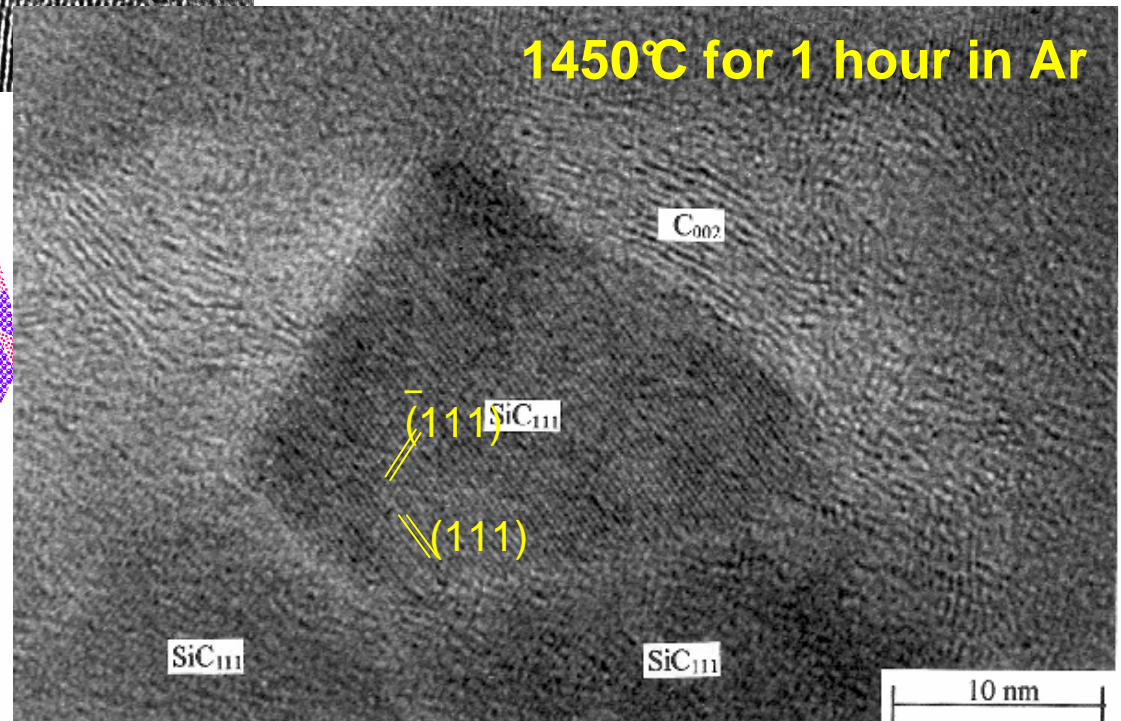
Sylramic

Microstructure of Hi-Nicalon fibre (0.5% wt O)

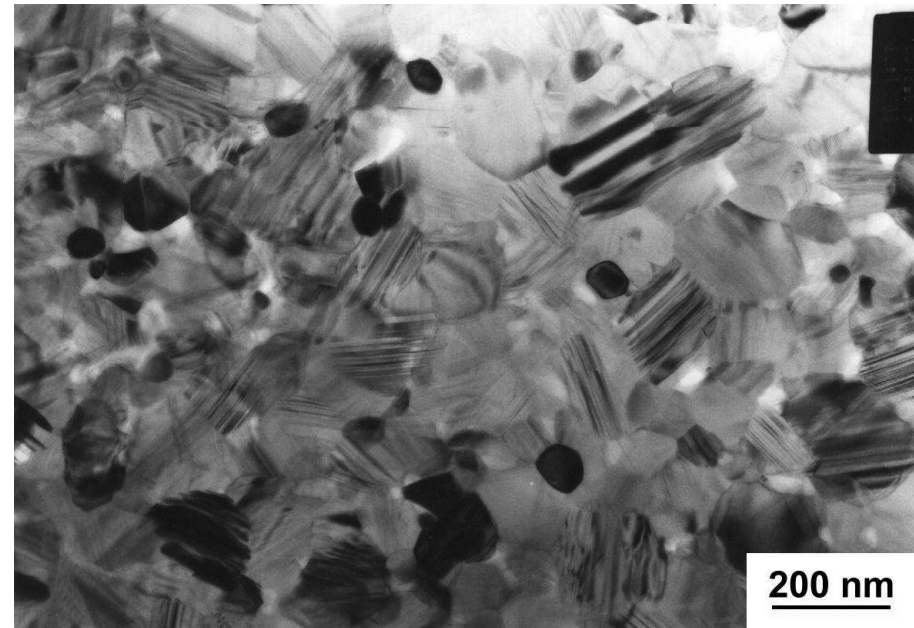
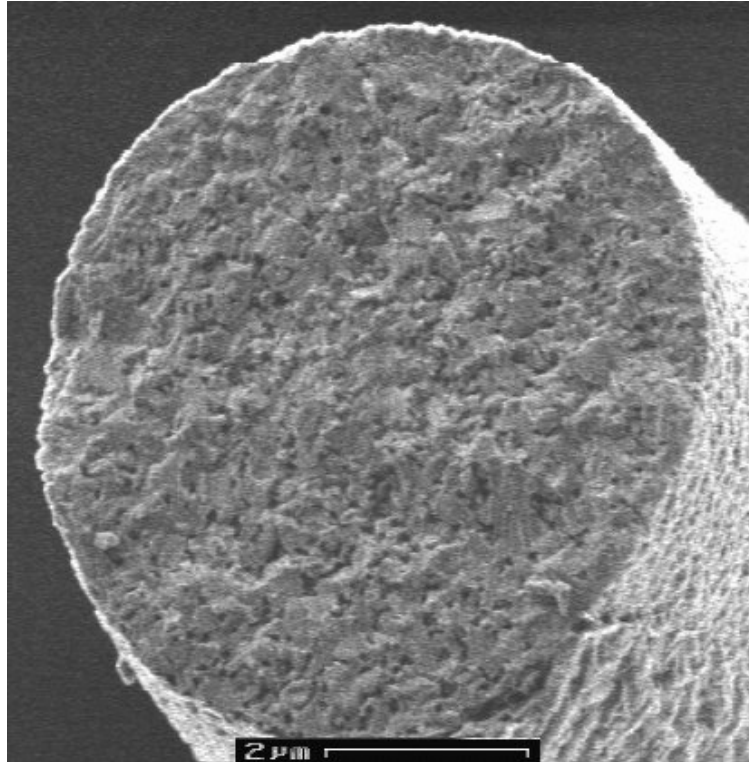


β-SiC 10 nm,
+ C 2 nm
+ Si-C intergranular phase

Higher stiffness: $E=260$ GPa
 $\sigma_R=2$ GPa
Chemical stability
Improved creep properties

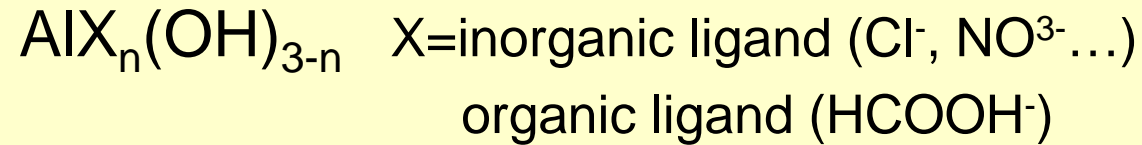


NEW SINTERED SiC FIBRES
“near stoichiometric” SiC fibres



Production des fibres d'alumine

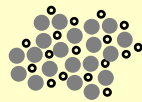
Filage d'un precurseur: sel d'aluminium



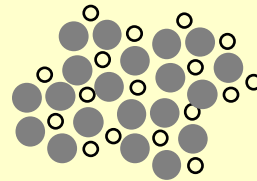
Pyrolyse

amorphe \rightarrow eta + gamma \rightarrow delta \rightarrow theta \rightarrow **alpha : stable**

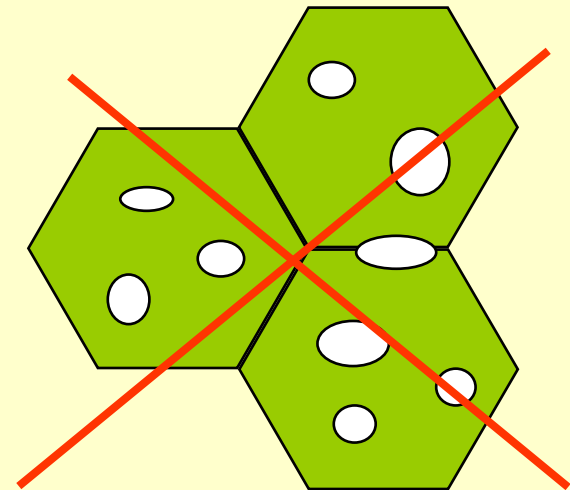
300-400°C



400-1000°C

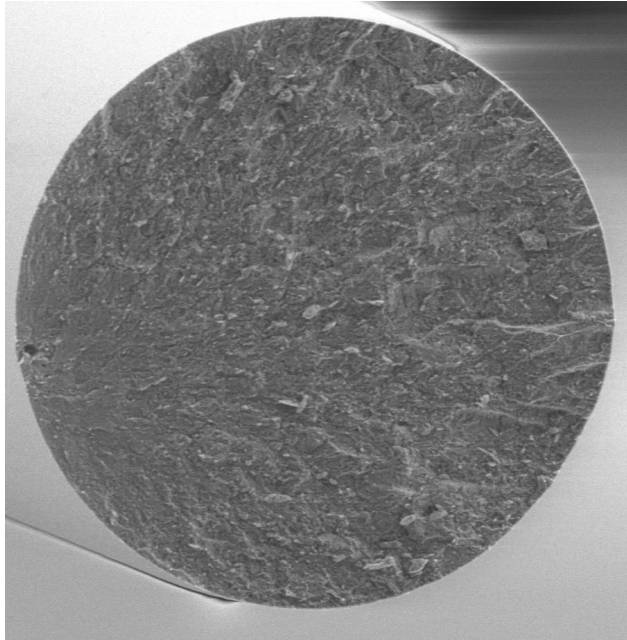


1000-1100°C

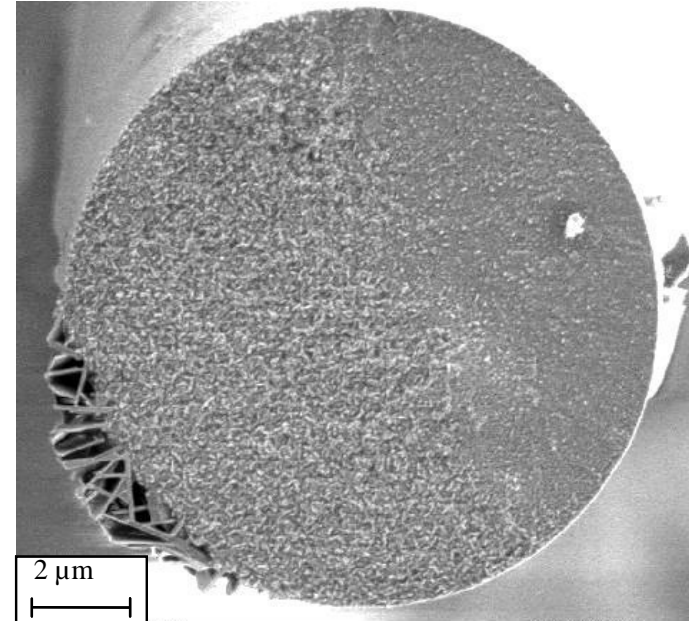


1 μm





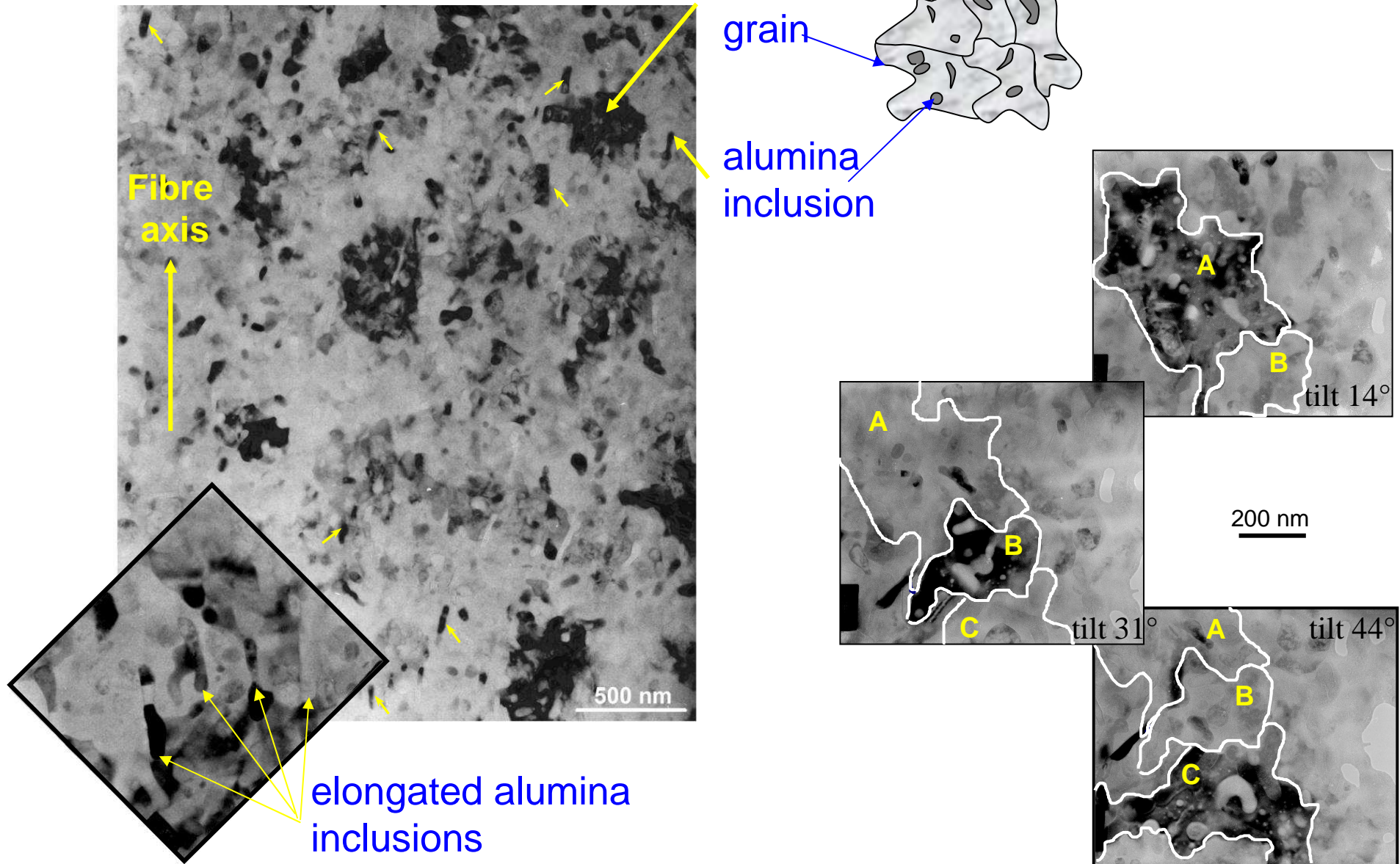
Fracture morphology of an alumina-mullite Nextel 720 fiber tested at room temperature



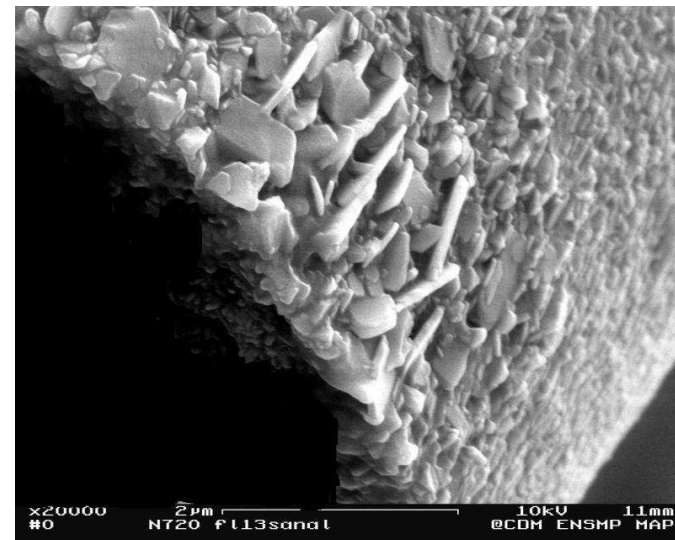
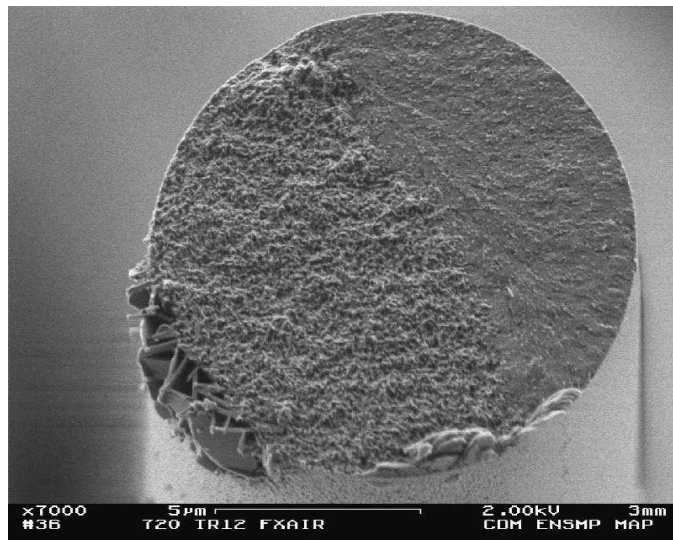
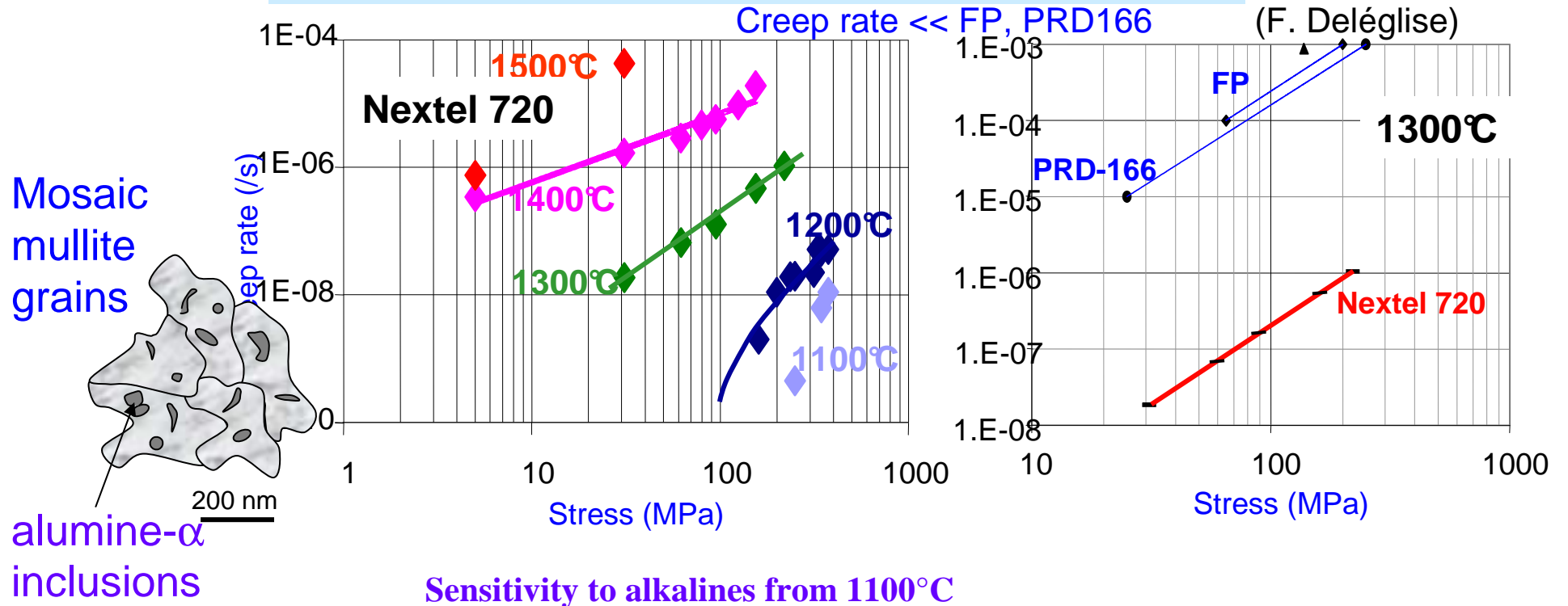
The effects of alkaline contamination on an alumina-mullite Nextel 720 fiber tested in tension at 1200°C

Nextel 720 : mullite α -alumina

(Thèse F. Deléglise)



Nextel 720 – Creep of bundles to 1500°C



Conclusions

Les structures renforcées par des fibres comprennent toutes classes de matériaux, du caoutchouc aux céramiques. Elles peuvent casser à cause de différents mécanismes. Pour les cerner correctement, il faut une compréhension de leur microstructure, souvent à l'échelle atomique, révélée par une variété de techniques dont la microscopie électronique à balayage est la plus importante.