UPMC - ESPCI - CNRS

RESULTS

Sciences et Ingénierie de la Matière Molle

Physicochimie des Polymères et des Milieux Dispersés

2007-2012

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GENERAL INTRODUCTION TO UMR ACTIVITIES

Introduction

We tackle soft-matter problems that are, in general, long standing engineering problems. They are associated with complex general and fundamental scientific questions and are of long-lasting interest for industry. The name of the lab was recently changed from “Polymer and Dispersed Media Physical Chemistry” to “Soft Matter Science and Engineering”. The reason for that change was to better reflect our scientific policy.

The subjects we deal with are, for instance, fracture, adhesion, friction or glass transition, and every-day life materials problems related to foams, gels, polymer nano-composites and cement. Despite the fact they have aroused interest for centuries for some of them, those subjects share complex underlying scientific questions. We aim at creating new perspectives on those topics, by developing original approaches based on the joint development of novel experimental techniques, new concepts and model systems.

Indeed, in order to be competitive on those widely studied topics, three strengths are required:

- Being original enough, developing multidisciplinary approaches with either new model systems or new experimental techniques, and introducing new concepts at the frontiers of the actual technical and scientific knowledge,
- Being able to combine long-term support from industry with academic support,
- Seeking and anticipating on problems that are relevant not only for industry, but that are also of high level scientific value.

In summary, our goal is to develop **a comprehensive approach**, based on clear physical chemistry and physics concepts, in order to address questions that constitute both **engineering and fundamental problems**. This is indeed one of the reasons for the success we meet in our collaboration with companies and in the academic dissemination of our results.

Indeed, all the topics we are interested in have applications in various industrial sectors: rubber and tires, biomedical, food, building materials and microfluidics to cite a few. Our studies are nevertheless only partially financially supported by industry, since we need a different framework to build new tools (systems/concepts/set-up) on new topics and to further demonstrate that our ideas are both scientifically challenging and of potential interest for industry. The early development of these studies indeed requires academic support. Accordingly, we have obtained long term industrial supports on topics such as adhesion, cement, tribology, nanocomposites but, conversely, we work only with academic support on microgels, hybrid hydrogels, photo responsive foams, and free surface fluctuations.

Scientific output

The activity report is divided into three themes:

- *Fundamental physics of soft matter*
  Friction, polymer constrained dynamics in nanocomposites, and free surface thermal fluctuations. This theme aims at bridging statistical physics and mechanics of disordered matter.

- *Soft polymer networks*
  Design of soft polymer network and gels, adhesion and fracture. This theme encompasses activities on the effect of changes in material structure on the mechanical response in complex loading situations. The materials studied are soft polymer networks and gels.
- *Tailoring interfaces and mesoscopic structures*
  Photoresponsive interfaces and polymer/colloids design and understanding. This theme handles questions relevant to the physical-chemistry of suspensions, emulsions and foams with a focus on interfacial properties.

For the sake of clarity, those three themes will be developed independently later in this report despite numerous overlaps both in terms of human resources and scientific questioning.

Main solved issues

The main questions that have been solved in the period of interest can be summarized as follows:

- We have developed foaming azo-benzene photo-surfactants. We have shown that conformation changes under illumination not only modify the interfacial energy but above all generate molecular fluxes and macroscopic flows.
- Most of the effects of polymer plasticizers for cements may disappear in the presence of aluminate. We have shown that large enough lateral chains of comb-like polyelectrolytes prevent the nefarious formation of aluminate/polymer hybrid materials.
- We have developed a new class of hybrid hydrogels. The mechanical toughness is ensured by the reversible adsorption of polymer chains on silica particles.
- We have demonstrated that confined polymers in nanocomposites exhibit both a gradient of temperature and a broadening of the glass transition. The broadening is responsible for most of the mechanical properties of car tires.
- We have demonstrated that local friction of a rough surface on soft materials is not governed by the Coulomb law, but by a power law relation between pressure and shear stress.

From 2007, the lab members have published 266 papers in international peer reviewed journals (see the full publication list in appendix). The range of scientific fields covered by the different journals is very large, thus reflecting the various backgrounds of the lab members: Based on ISI web of science, 99 of our papers fall in the category Polymer Science, 81 in Physical Chemistry, 78 in Materials Science, 37 in Chemistry and 35 in Physics. Although we have not published in high impact interdisciplinary journals, we have published in journals varying from Journal of Materials Chemistry or Angewandte Chemie int. edition, to Physical Review Letters and Europhysics Letters. When targeting a specialized audience, we have also published in the most respected journals in the field (for instance Cement and Concrete Research for cement, Review of Scientific Instrumentation for technical developments, Journal of Magnetic Resonance for NMR, …). Indeed, a given project usually leads to publications in journals covering different fields, corresponding to the different steps of the project; for instance, those steps may be the definition and characterization of an original model system, followed by the measurement and the interpretation of its macroscopical properties, possibly using a new instrument or technique.

Academic influence and attractiveness

The lab members have developed numerous collaborations either at the national or international levels. As a consequence, 75% of our publications are co-authored with members of other groups. Half of them are written in collaboration with foreign researchers and/or or researchers from private R&D labs. On the academic side, most national collaborations are funded by the ANR. Among these

collaborative research works, lab members were involved in 17 ANR projects and two young researcher (JCJC) ANR projects (7 of them are coordinated by lab's members). Lab researchers have also been involved in various European projects (3 research projects and 3 academic networks) gathering both industrial and academic partners.

International joint projects represent one third of all collaborative works and also one third of published works. Their development is especially facilitated by a large number of invited scientists stays (20 renowned personalities visited in the lab, from one month to one year). These invited professor positions were funded either by industrial (Saint-Gobain, Michelin, Total) or academic chairs (UPMC, ESPCI).

The lab is also attractive for students. Student recruitment is facilitated by the strong links which exist between the lab and both UPMC and ESPCI. About 6 PhD students are joining us each year (their background is 1/3 from ESPCI, 1/3 from other "grandes écoles", 1/3 from universities). Furthermore, the numerous contacts we have developed with companies help our students to easily find a position once they quit the lab. As a matter of fact, a large proportion of those who wish to work in private companies are hired before their PhD defense.

Dissemination to industry and to the general public

As mentioned earlier, one strength of the lab is the great number of industrial collaborations which is reflected by the numerous research contracts. Generally, these collaborative works with industry include the funding of theses (41% of the theses are funded by industrial companies or under CIFRE).

Several members of the lab have also responsibilities in 3 industrial chairs hosted by ESPCI (Michelin, Saint-Gobain and Total). They are in charge of selecting students for fellowships and of invitating foreign scientists. We largely benefit from this activity. In the same way, we are involved in scientific and administrative tasks in several organizations: scientific dean of ESPCI, membership of the scientific councils of Elastopôle, Saint-Gobain, Solvay-Rhodia, Dutch Polymer Institute... Moreover, we have acted as consultants for companies (about one hundred days for French and foreign companies).

Dissemination of our scientific output to the general public is reflected by the participation of some of the lab's members to the exhibitions and conferences hosted by 'Espace des Sciences' whose aim is to disseminate science to the general public. Similarly, several of us participated to scientific TV shows (E=M6,« C'est pas sorcier », Nuit des Chercheurs).

Training of young researchers

As mentioned previously, the lab welcomes a large number of students each year, Ph.D students and post-doctoral fellows, but also master students, bachelor students and students from the classes préparatoires within the framework of TIPE projects, and even middle and high school students ('collège' and 'lycée' internships).

Each year, lab members supervise about 8 internships involving 3rd year ESPCI students. These internships often provide unique opportunities to explore new ideas or experimental approaches within the framework of short term projects.

Two thirds of the permanent researchers are UPMC or ESPCI faculty members who are required to devote half their working time to teaching. In addition, all the lab CNRS researchers have teaching activities either at ESPCI or UPMC. Several lab members are in charge of teaching programs ("spécialité" Material Science of the master "Chimie Paris Centre", Licence Professionnelle Industrie Chimique et Pharmaceutique, spécialité « Métiers de la Chimie de Formulation ». One of us has

largely contributed to the creation of a new master track: “Physical Mechanics” in the master 2 “Complex Systems”. The lab contributes largely to various training programs and short courses targeted at industry and young academics (Ecole des Houches, formation continue UPMC, POLYMAT, Collège Polytechnique…).

As a part of their training, PhD students and post-doctoral associates are encouraged to directly supervise interns or to be responsible for the maintenance of a lab equipment. Scientific discussions between students occur in particular during monthly meetings in which they debate over oral presentations given by two students. They also organize with 4 others IMPC (Institut des Matériaux de Paris Centre) laboratories a common meeting twice a year focused on materials science. Additionally, once a year 2nd year PhD students and post-docs present their research activities to the laboratory over one half day. We also encourage our students and post-docs to attend international conferences and nearly all of them have attended at least one or two international conferences before they graduate or leave the lab. To favor dissemination of results, each year, several of our PhD students write their thesis in the English language.

Most of the PhD graduates of the lab find a job in companies (85 %) and are frequently hired before their PhD defense (Michelin, Rhodia, Saint-Gobain…). Several of our PhD students join the academic research.

FUNDAMENTAL PHYSICS OF SOFT MATTER: FRICTION AND GLASS TRANSITION

Introduction

Friction and glass transition are long standing problems in soft matter physics that are also of major practical interest. An increasing number of theoretical works – sometimes contradictory – attempt to address those problems; however, experimental studies on those topics became scarce in the past decade, as a result of the failure of most experimental approaches to provide new insights into these phenomena. The challenge was thus to be able to obtain sufficiently new experimental results in order to renew the underlying soft matter physics concepts. For both friction and glass transition, we have developed a long term approach built from the design of new experimental tools. In what follows three activities at different completion stages are detailed: two of them (glass transition gradient in filled elastomers and soft materials friction) were initiated a decade ago and have successfully provided a new understanding of fundamental concepts, the third one (dynamics of colloidal systems close to the glass transition) has been developed more recently.

State of the art and international academic context

In the physicist community, friction has been tackled using either macroscopic experiments or nanoscale tools such as scanning probe microscopy (SPM) or surface force apparatus (SFA). All these approaches do not pay much attention to the details of the strongly non homogeneous stress and displacement fields, which are invariably achieved within frictional contacts. However, such aspects are fundamental in the understanding of a variety of problems such as the friction of rough surfaces or stick-slip instabilities. We were the first to measure spatially resolved stress field distributions within sliding contacts and to apply this approach to the friction of rough surfaces, a topic which is traditionally dominated by theoreticians (B. Persson in Europe, M. Müser or M. Robbins in the USA).

In the last two decades, glass transition has been the object of a renewed interest with the finding that confinement is able to modify the glassy dynamics of polymers. A dozen teams in the world have consequently devoted their research to confined polymers dynamics in thin films. We first suspected that such effect could be involved in the properties of nanocomposite polymers; and were also the first to perform precise measurements on the confinement effects using our model system. Owing to the difficult synthesis of model systems, our competitors (among which Kumar and Colby in the USA and Colmenero and Richter in Europe) started similar studies much later (ca 2005).

Thanks to both the French organization of research and the support of our industrial partners, we have been able to conduct long term projects, a large part of which was devoted to the development of original tools. Those tools are either “physical tools” consisting in novel experimental set ups, or “chemical tools” consisting in model systems. As a result, we are now the only laboratory in the world able to measure highly spatially resolved stress fields within frictional contacts, as well as to design models for filled elastomers in which the polymer confinement and the physical chemistry are extremely well-controlled. We are thus among the leading research teams in the competition for the physical understanding of friction and glass transition effects. More recently, we have developed an extremely light and efficient optical tool to characterize the mechanical properties of soft matter systems. Based on the measurements of thermal fluctuations to make the systems “self-revealing”, this tool will hopefully provide new insight into glass transition in colloidal systems.

International influence and attractiveness

We have several collaborations, with internationally recognized scientists in each domain, R. Colby (Upenn) for glass transition, K. Saalwaechter (Halle) for NMR measurements of nanocomposites, D. Long (CNRS/Rhodia) for theory of confined glassy polymer dynamics, A. Prevost and G. Debrégeas (LPS, ENS) and E. Barthel (SVI CNRS/Saint-Gobain) for friction, A. Lyulin, (Eindhoven) for the molecular modelling of friction. Thanks to our optical tools we are also starting a collaboration with W. Poon (Edinburgh), expert in the field of colloidal glassy phases. The capabilities of our contact imaging tools have also motivated ongoing collaborations with M. Sitti (Carnegie Mellon) in the field of biomimetic adhesives and with N. Spencer (ETH Zürich), J.P. Gong (Hokkaïdo University) and M. Chaudhury (Leighigh University, USA) in the field of friction of patterned surfaces.

Insertion in the general orientations of the laboratory

Fundamental physical issues on glass transition or friction are some of the questions addressed in the theme. They are discussed by using original tools, model materials and innovative experimental techniques. As for most of our studies, the physical chemistry characteristics of the samples are explicitly taken into account and exploited to strengthen the obtained descriptions. Despite the rather fundamental character of the topics, most studies are developed with industrial partners. In these respects they are typical of the lab approach.

Societal relevance

Although our tools have been developed in order to better understand fundamental problems, they are also well adapted to solve industrial questions. Five research contracts with major international firms such as Michelin or Saint-Gobain have been completed or are still running.

Training of young researchers

The multiple connections we have in both the industrial and academic worlds are attractive for students: in the past five years, 7 master 2 students, 5 PhD students (among which 3 defended their thesis) and 9 post-doctoral students have worked or currently work on those topics. All our former Ph. D students are now employed in industry. Among our former post-doctoral students, one has an academic position (maître de conferences), one has a position in industry, and four are post-doctoral fellows in a foreign country. All the permanent researchers teach or have taught in master courses. We are in particular strongly involved in the masters “Science des Matériaux et Nano-objets” (responsible for the UE “Propriétés Mécaniques des Matériaux”), and “Chimie et Physico-chimie des Matériaux” (responsible for the UE “Propriétés Mécaniques des Matériaux Polymères”).

Solved questions

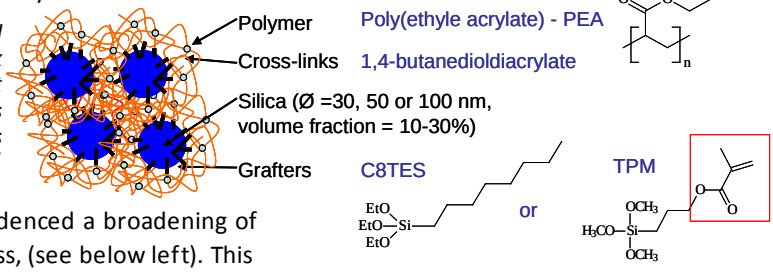
- We have proved that in nanocomposites, the polymer exhibits a gradient of its glass transition temperature. We have measured it by different methods, and obtained a quantitative agreement between results. We have also evidenced a broadening of the glass transition width. These results provide a quantitative explanation for most of the mechanical properties (either linear or non linear, provided that the strain remains of small amplitude) of nanocomposites [164,171,207,217,252,253,254].
- We have shown that in a smooth and single asperity contact, the friction stress does not depend on the normal force. Within rough contact interfaces, we have evidenced that the local friction stress scales with the contact stress as a power law that depends on the details

of surface roughness. These results should serve as a basis for the discussion of theoretical models for friction of rough surfaces [63,148,215,235,250].

- We have developed an optical apparatus that uses the measurement of surface fluctuations to provide a mechanical characterization of soft matter systems. We have shown that the apparatus provides a new route to evidence dynamical heterogeneities close to the glass transition, and we are currently using it to provide new insight in glassy colloidal phases [91, 123, 221, 256].

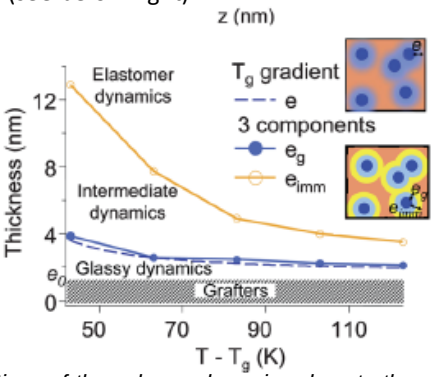
FILLED ELASTOMERS : REVEALING DYNAMICS OF CONFINED POLYMER NEAR THEIR GLASS TRANSITION

Since 1997, we develop a comprehensive understanding of the mechanics of filled elastomers. For that purpose, we have elaborated model filled systems consisting of monodisperse silica particles with various diameters embedded in a polymer matrix. The synthesis procedure ensures an intimate contact – and optionally a covalent bonding- between the matrix and the nanoparticles. The dispersion of the particles – and thus the degree of confinement of the polymer - is controlled and characterized by neutrons scattering. We have measured by NMR the dynamics of the α relaxation process in the vicinity of the particles. We have evidenced the existence of a gradient of glass transition temperature which also affects the calorimetric response of the system.



Model samples consist in silica spherical particles grafted within an elastomer matrix using either a non covalent or a covalent grafter. The quality of the dispersion is ensured by using the acrylate monomers as a solvent of the stable silica colloidal suspension.

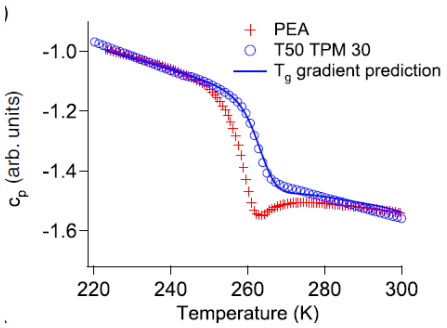
In addition to this effect, we have evidenced a broadening of the low frequency side of the α process, (see below left). This additional slowing down of the chain dynamics governs the mechanical properties of the filled elastomer when it affects a polymer thickness of the order of the distance between neighboring particles. For instance, particles connected by polymer domains with modified dynamics create a network that can be strain-softened under small amplitude oscillatory strain. Referred to as the Payne effect – this phenomenon is quantitatively linked to the connectivity of this network. We show that all the experimental mechanical results with various filled systems collapse semi quantitatively on a single master curve (see below right) relating the amplitude of the non-linear response to the average connectivity of the network (see below right).



Modifications of the polymer dynamics close to the surface of the silica particles as a function of the distance to the glass transition temperature. Blue curve indicates the thickness of the polymer layer with a gradient of glass transition temperature, yellow curve the thickness of polymer layer affected by the broadening of the glass transition.

H. Montes, F. Lequeux, F. Casas (PhD), T. Chaussée (PhD), A. Papon (PhD)

- Publications : [164,171,207,217,252,253,254], 5 invited conferences 5
- Support: Rhodia, ANR Taylrub, ANR-DFG Dynafil
- Collaborations : LPMA (D. Long, L. Odoni) Lyon, K. Saalwaechter, Halle Germany , J. Oberdisse (Montpellier)



Calorimetric response of the pure polymer (crosses) and of the filled elastomer (circles). The continuous line is the prediction obtained from the NMR measurements thanks to the Tg gradient model, without any adjustable parameter.

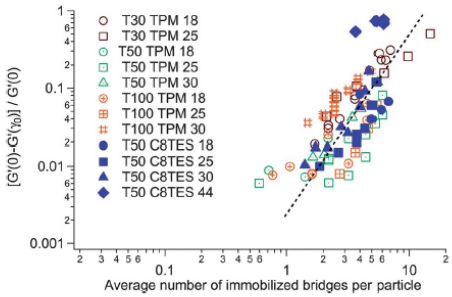
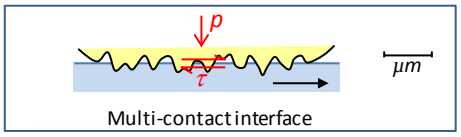


Fig. 6 Amplitude of the Payne effect as a function of the average number of immobilized bridges in various samples and several temperatures between 6 °C to 70 °C. The line is a guide for the eyes.

Amplitude of the non linear mechanical response as function of the connectivity of the particles within the network defined by the polymer layer with broadened glass (yellow curve in the figure on the left). The particles connectivity is computed both from NMR and neutrons scattering experiments. Symbols correspond to various samples with covalent grafters, different silica diameters, silica volume fractions, and temperatures.

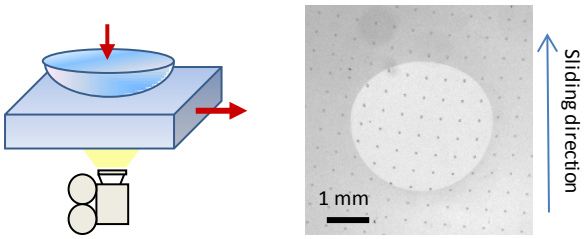
LOCAL FRICTION OF RUBBERS WITH ROUGH SURFACES



Multi-contact interface

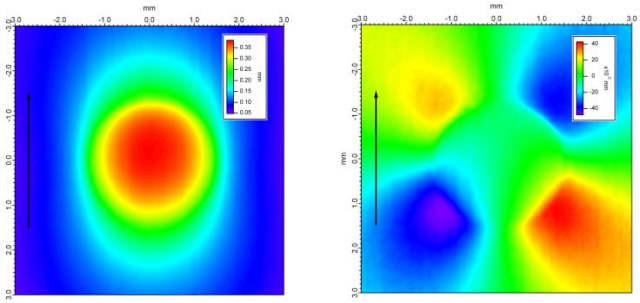
Friction of rubber with rough surfaces involves a poorly understood interplay between contact mechanics issues and the physics of dissipative processes at the asperity scale. This problem can not be adequately tackled using integrated quantities such as friction force measurements due to contact non homogeneities. To overcome these limitations we have developed an approach based on the measurement of spatially resolved displacement and stress fields from the resources of contact imaging.

Contact imaging



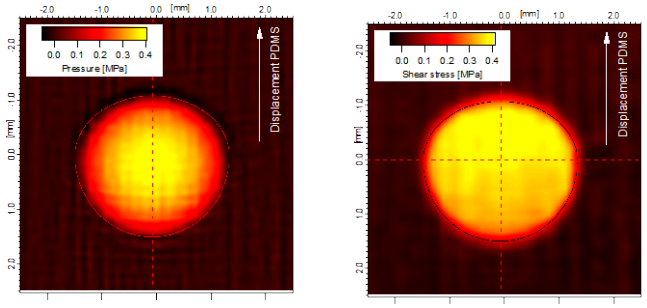
A surface marked transparent silicon rubber (PDMS) is rubbed against a glass lens which can be made either smooth or rough. Surface displacement of the rubber substrate are measured from markers displacements. Image accumulation under steady state sliding allows to achieve a good (~ 10 μm) spatial resolution.

Displacement field



Surface displacements are measured under steady friction. The displacement components are determined along (left) and perpendicular (right) the sliding direction.
(The PDMS substrate is moved from bottom to top with respect to the fixed glass lens).

Contact stresses



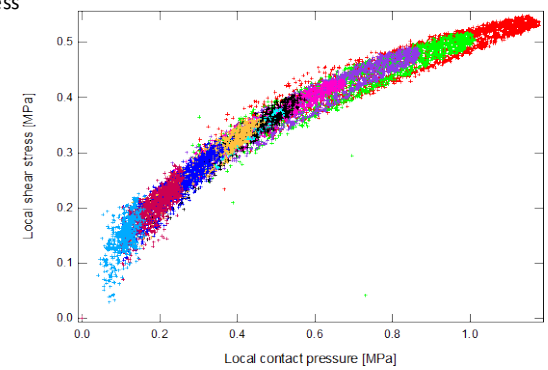
Contact pressure

Surface shear stress

Inversion

The measured displacement field is inverted in order to retrieve the corresponding contact pressure (left) and surface shear stress (right) distributions within the contact. The developed numerical inversion procedure takes into account both the geometrical and material non linearities associated to the occurrence of finite strain at the surface of the rubber.

The local friction law, i.e. the relationship between contact pressure and frictional stress is determined. Within rough contacts, a non Coulombic, power law dependence is identified. In the case of a smooth glass substrate a pressure independent shear stress is measured.



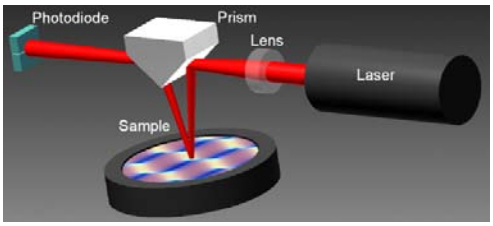
Local friction law giving the dependency of the local shear stress on the actual contact pressure for a contact between a smooth rubber and a statistically rough glass surface (r.m.s. roughness ≈ 1 μm).

A. Chateauminois, C. Fretigny, D.T. Nguyen (PhD), P. Paolino, M.C. Audry (Post-docs)

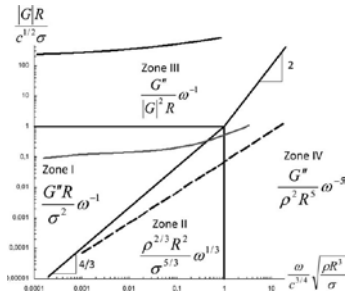
- Support: ANR Dynalo, Michelin (CIFRE) , Saint Gobain (Post-doc).
- Publications : [63,148,215,235,250], 5 invited conferences
- Collaborations: A. Prevost (LJP, ENS Paris), E. Barthel (SVI Saint Gobain), N. Spencer (ETH Zürich) and J.P. Gong (Hokkaïdo University).

SURFACE FLUCTUATIONS OF COMPLEX MEDIA

We have developed a new non invasive tool to study in or out of equilibrium complex media: Surface Fluctuation Specular Reflection (SFSR) spectroscopy, based on the principle that surface fluctuations self reveal a medium.

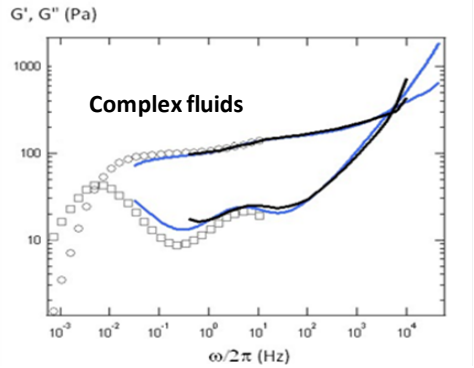


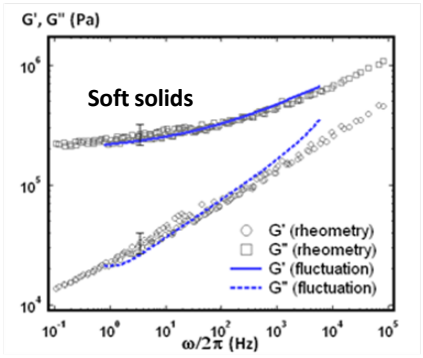
Schematical representation of the optical set up we have designed to measure the thermally induced waves at free surfaces. We use the specular reflection of a laser beam that is focused on the free surface of the medium. The fluctuations in the position of the reflected beam are related to the surface fluctuations. The radius of the laser beam at the surface sets the characteristic lengthscale of the measurement.



We have shown that, in a viscoelastic modulus vs. frequency diagram, the measurements explore different regions, in which the leading effects ruling the propagation of thermal waves differ: surface tension, inertia, elasticity and/or viscous dissipation may dominate.

A new method for bulk linear viscoelasticity measurements

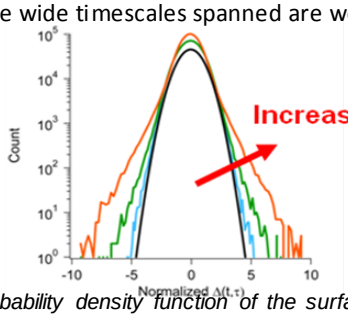




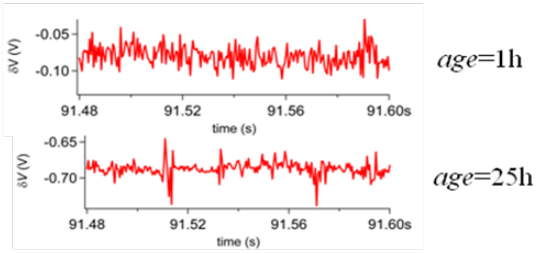
We demonstrated that surface fluctuation measurements provide **a high bandwidth** (up to 6 decades in frequency), **non invasive** characterization of the bulk linear viscoelasticity of materials ranging **from low viscosity liquids** to **soft solids**, either transparent or not.

A tool to study the aging dynamics of colloidal glassy systems

Using surface fluctuation measurements, we have evidenced highly intermittent events occurring during the aging of a glassy colloidal phase. The mesoscopic lenghtscale at which the surface is probed, as well as the wide timescales spanned are well suited for the study of dynamical heterogeneities of colloidal glasses.



Probability density function of the surface fluctuations of laponite suspensions becoming non Gaussian as the suspension ages.



Recorded signal as a function of time: as the colloidal phase ages, dynamical heterogeneities (large and fast events) appear.

C. Frétigny, F. Lequeux, L. Talini, E. Verneuil, B. Pottier (PhD), A. Raudsepp, A. Deboeuf (post-docs)

- Publications: [91, 123, 221, 256]
- Support: Research in Paris (Ville de Paris), Saint Gobain.
- Collaborations: J.-P. Roger (Institut Langevin, Paris), D. Fournier (LPEM, Paris), J.-F. Palierne (ENS Lyon), W. Poon (U. Edimburg).

Objectives for 2019

Although our long term projects are now completed, several short term projects or developments of the past studies are planned for the coming years.

Among the completed projects, experiments on model filled-rubbers have provided most of the experimental answers we expected. They have also evidenced the fact that dynamical heterogeneities are the keys for a further understanding of the mechanical properties of confined polymers. We therefore plan to study blends of miscible polymers, in which the dynamical heterogeneities are significantly amplified.

Several projects are related to the study of friction between solid surfaces: first, the experimental link between surface roughness and friction will be quantified through the use of patterned surfaces with well mastered topographical characteristics; second, the influence of viscoelasticity of the medium will be elucidated; third, stiction phenomena (transition from static to dynamic friction) will be investigated, using the same methodology as for steady-state friction processes for the understanding of transient phenomena.

Finally, two projects involve surface fluctuation measurements: one deals with aging of a glassy colloidal phase (what is the local dynamics?), and the other considers confined systems, such as thin viscoelastic films (what are the effects of confinement?).

Conclusion

In conclusion, we have developed an original approach to address complex fundamental questions on the physics of soft matter; the long term studies we have conducted were based on the development of new tools, either instrumental or chemical. The related works have been especially fruitful in terms of scientific production, relationship with the industry and student formation. In the next years, most of the involved researchers will further develop those topics, but an increasing part of their time will be devoted to the new projects described in the relevant sections.

Involved researchers

A. Chateauminois, M. Ciccotti, G. Ducouret, C. Frétigny, J.-L. Halary, F. Lequeux, H. Montes, L. Olanier, L. Talini, E. Verneuil.

Scientific output

19 invited conferences, 90 publications in international journals, 1 patent

Ressources

5 Ph. D. Students:
CIFRE fellowships: T. Chaussée (Rhodia), D.T. Nguyen (Michelin), A. Papon (Rhodia), P. Shi (Michelin).
University fellowship: B. Pottier (UPMC)
9 Post-doc students: M.-C. Audry (Saint-Gobain), A. Debeuf (ATER ESPCI), T. Kajiya (Contrat), E. Janiaud (DPI), P. Paolino (ANR), L. Raboin (ANR), A. Raudsepp (Ville de Paris), M. Trejo (ANR), D. Yang (ANR).
7 Master 2 students
Funding: ANR Dynafil, ANR Tailrub, ANR Dynalo, Research in Paris

Industrial contracts: Rhodia, Michelin, Dutch Polymer Institute, Saint-Gobain

SOFT POLYMER NETWORKS

Introduction

Soft materials have always been used in industry, as adhesives or rubbers, and in biomedical related sciences. The mechanics of soft materials is complex, including large strain, non-linear elasticity, viscoelasticity and plasticity. Yet soft polymer materials are often polymer networks and their design relies heavily on polymer science at the frontier between liquids and solids. In this field, our scientific approach is to combine physics, chemistry and mechanics as tools to develop new materials or to better understand the properties and function of existing ones. We work with model materials and surfaces and we are specialized in non standard characterization techniques for situations where important deformation gradients are present such as adherence and fracture of soft materials. Our methodology combines in-situ structure and/or dynamics investigations with a quantitative measurement of the mechanical properties.

State of the art and international academic context

Adhesion and mechanics of soft and viscoelastic materials and hydrogels is an area where the academic community is relatively small but which is of very high industrial relevance and in the case of wet materials is very important in life sciences. It is clearly a multidisciplinary area and the international competition tends to be spread over several fields (physics, surface and materials chemistry, mechanics, biology) and remains within its specific field. Few well identified groups (Gong at Hokkaido, Suo at Harvard and Shull at Northwestern) have a broad and comprehensive approach of these problems from a truly multidisciplinary prospective. Many groups worldwide (Ito and Haraguchi in Japan, C. Frank at Standford, O. Okay in Turkey) focus their research on designing new soft materials or surface modification methods to obtain enhanced properties (adhesion, toughness, conductivity at low percolation) but using relatively simple characterization methods, or on characterizing with sophisticated techniques relatively simple models systems (Baumberger in Paris, Fineberg in Israël).

We try to do both: identify structural or molecular features of the materials which have a significant influence on macroscopic properties and systematically investigate the effect of a change in material parameters on mesoscopic structure and material properties using advanced techniques. To be effective, this multidisciplinary approach requires close collaborations between scientists with complementary expertise (physics, chemistry, and mechanics) and a shared common research objective. This type of collaborations is rare within the same group. We face two types of competition: on the chemistry side several talented polymer chemistry groups have developed new materials or thin films with improved properties but often lack the expertise to carry out detailed investigations on the underlying reasons for the improved properties. On the mechanics or physics side there are many excellent groups but they typically lack knowledge of materials science and interest in truly investigating materials effects. Many new interesting concepts have appeared in physics and mechanics or nanocomposites in recent years and beg to be refined and improved with a true diversity of material chemistries and innovative techniques.

International influence and attractiveness

In the area of adhesion, whether pressure-sensitive-adhesives or hydrogels, we capitalize on years of experience and are clearly world leaders. Our key competitive advantages are the combination of industrial knowledge (from extensive consulting) with high level science in this area and a truly multidisciplinary approach combining non standard chemistry (outside collaborations or in-house)

with unique characterization methods. This gives us a very high visibility in this area as attested by 12 adhesion related invited conferences, two international scientific prizes and two European collaborative projects.

In the hydrogel and rubber area which is quickly expanding (14 invited conferences), our visibility increases and we have found our niche in the use again of high level interdisciplinary science, controlling model systems with chemistry, polymer dynamics in the gel with light scattering methods and macroscopic properties with large strain mechanical tests. Our competitive advantage is here. We focus on materials science rather than mechanics, i.e. we use and in some case develop, advanced techniques such as IR and Raman spectroscopy, SAXS and SANS, custom-made adhesion testing, neutron reflectivity to understand why some materials display very enhanced properties and we are able to vary material parameters through synthesis in our model systems.

Overall, our approach and project on soft materials has gained considerable international visibility during this period as shown by the numerous invited lectures in international conferences worldwide and by the international collaborative projects and foreign post-docs applying to work on our research projects.

Insertion in the general orientations of the laboratory

Designing materials for specific macroscopic properties requires a deep understanding of their underlying mechanisms. In turns, it imposes to work on simplified models, which usually necessitates originality in the chemistry. The projects gathered in this section are typically representative of this approach. The interplay between industrial questions, design of model systems and fine analysis of the properties takes advantage of the different strengths of the lab.

Societal relevance

We work on a variety of projects many of which are inspired by industrial problems but are simplified to be tractable on a more fundamental sense. We try to achieve a good balance between direct industrial collaborations, ANR funded research projects and European research projects to build a bridge between application and fundamental knowledge. Several of our projects are connected with sustainable development (materials made from water-based emulsions) and biomedical applications (self-assembling of polysaccharides). C. Creton is the Scientific Chairman of a technology area of the Dutch Polymer Institute, which funds industry/academia partnerships.

Training of young researchers

Three members of our group are deeply involved in teaching at the bachelor and masters level in polymers and soft matter formulation at University Pierre et Marie Curie and at ESPCI ParisTech. At the Bachelor level, we are in charge of a professional orientation in the field of formulation industry and our group is very active in teaching, supervising students during their 6 weeks internship and connecting with industrial partners where students make their alternating apprenticeship. In the framework of the master's degree, we (UPMC, ESPCI and CNRS members) are strongly involved in the second year education, supervising the polymer science curriculum at the master's level, giving lectures in various teaching units and supervising several master internships. For a French group we supervise a large number of students, for short internships of less than three months (typically 5-8 per year), master's level projects (3 per year typically) and at the PhD level (1-2 per year typically). During the period we have graduated 9 PhD students (2 foreign students) in this area: 3 have now maître de conferences positions, 5 are engineers in industry and one is post-doctoral fellow in

Germany. We have supervised 21 post-docs (5 foreigners) of which 11 are now R&D engineers in industry, 4 are maîtres de conferences, 2 are Ingénieur CNRS and 3 are post-doc.

Solved questions

Apart from the three highlights presented below we have obtained the following important results:

Cavitation in rubbers: By developing a new methodology and carrying out temperature dependent experiments, we have shown conclusively that the cavitation strength of unfilled rubbers loaded under a triaxial stress is more controlled by the fracture toughness in mode I loading than by the elastic modulus of the elastomer [151,191].

Acrylic-Urethane Hybrid Networks for Soft Adhesives: Collaborating with two synthesis groups in Lyon and San Sebastian we proposed new design guidelines for new acrylic-urethane hybrid polymer networks for adhesive applications, synthesized by environmentally-friendly miniemulsion polymerization. [192,209,210,231,242]

Soft-Soft nanocomposite: We have proposed the concept of a soft-soft nanocomposite where heterogeneities in structure are controlled at the nanoscale between two soft phases [112,113,114].

Modeling of large strain viscoelastic materials: We have proposed a new constitutive model combining non-linear fluid viscoelastic behavior with finite extensibility of rubbers [114].

Adhesion Mechanisms : In collaboration with the group of A. Lindner at ESPCI, we have shown that the transition from interfacial failure and fibrillar failure with energy dissipation is controlled by the reduced parameter tan δ/G' and have studied instabilities during debonding. We have also measured the contact angle of a propagating contact line in a highly viscoelastic fluid/solid [82,167].

Carbon nanotubes in elastomers: We have developed a new and versatile method to disperse CNT in different elastomers. In all the investigated elastomeric matrices, considerable improvement in the elastic and tensile moduli have been obtained with the nanotube as well as an extremely low percolation threshold for electrical conductivity (0.05 wt% of CNT) [4,22,57,104,237].

Spectroscopy for interfacial CNT/matrix adhesion: We showed that Raman spectroscopy is an invaluable tool to understand many fundamental aspects of carbon materials and in particular the CNT/matrix interfacial adhesion [264,238, 239] .

Responsive macromolecular assemblies in bulk and at interfaces: We have shown that thermal and pH-sensitivity could be readily cross-coupled within the same system giving rise to macromolecular system with enhanced sensitivity. We have shown that it was possible to drive reversibly with temperature the formation of interpolyelectrolyte complexes (IPC) in bulk and at interfaces demonstrating to trigger macroscopic transitions like gelation or adsorption respectively [183,225].

Responsive bio-compatible polymers: Using alginates as water-soluble backbone we have shown for the first time that cold gelation could be driven with monovalent ions like potassium instead of multivalent ones. Moreover, by chemically modifying polysaccharides with thermoresponsive or hydrophobic side-chains we have developed biocompatible self-assemblies forming 3G-hydrogels or nanoparticles under a controlled environment [77,159,263] **.**

FRACTURE OF RUBBERS: DAMAGE MECHANISMS AT THE CRACK TIP

Filled rubbers have been used as tough and yet deformable materials for years in industrial applications such as tires or vibration dampers. Although semi-empirical strategies have led to excellent material properties, energy savings concerns have now pushed research beyond existing solutions and toward a more knowledge-based approach. One of the properties that need to be understood are the parameters controlling crack propagation in fatigue conditions, i.e. small amplitude deformations and a large number of cycles.

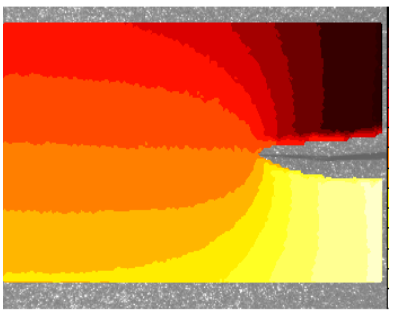


Figure 1 : Displacement field near the crack tip of a filled rubber as measured by digital image correlation.

We have started this project in 2007 and took a close look at the mesoscale (1-100 µm) close to the crack tip. In a first stage we used digital image correlation to measure the strain fields near the crack tip of a propagating fatigue crack in a series of model elastomers (figure 1). While the crack tip strain field on a single material showed predictably the presence of a strain singularity, the most interesting result came from the comparisons between different materials with varying filler contents and crosslinking densities. When the crack propagation per cycle is plotted as a function of the applied energy release rate G, large differences are seen between materials. A close analysis of the crack tip strain fields showed that the strain fields were very different for the same applied displacement boundary condition. Inspired by what is done for glassy polymers, we proposed the definition of a local energy release rate g_{local} and were able to rescale all crack propagation data on a single curve for the five tested materials suggesting that it is the local mechanics near the crack tip rather than an intrinsic bond resistance that controls crack propagation rate [214]. In a subsequent study in collaboration with E.J. Kramer at UCSB, we also showed for the first time that nanocavitation was an important damage mechanism at the crack tip. The cavities (20-50 nm in size), were clearly observed by real-time SAXS above a threshold stretch in uniaxial elongation by an analysis of the scattering invariant Q and of the scattering pattern (figure 2 and 3) [265]. The volume fraction and shape of the cavities was extracted from the scattering data and provided clear evidence of damage mechanisms occurring at the crack tip, where a fibrillar fracture is observed.

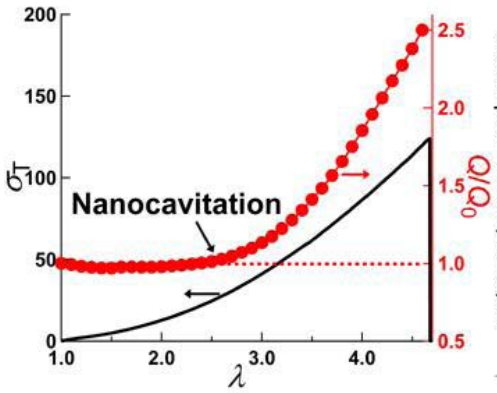


Figure 2: true stress σ_T and scattering invariant Q/Q_0 as a function of stretch λ showing the onset of cavitation.

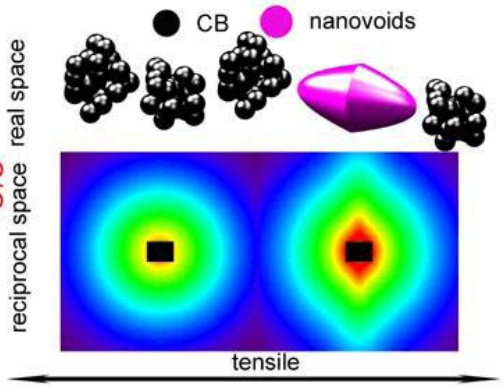


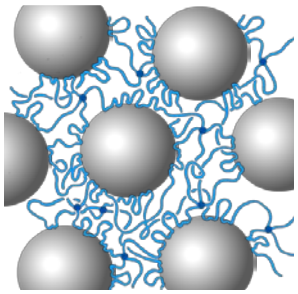
Figure 3: Schematic of filler particles and nanovoids geometry (above) and SAXS scattering pattern in the unstreched state (left) and at $\lambda = 4$ (right).

C. Creton, A. Marcellan, SamyMzabi (PhD), Jordan de Crevoisier (PhD), AntonellaCristiano (PhD), Huan Zhang (post-doc)

- Publications: [151,191,214,265], 6 invited conferences
- Support: ANR AMUFISE, DSM, Michelin
- Collaborations: E. Kramer (UC Santa Barbara), S. Roux (LMT Cachan)

HYBRID HYDROGELS : A VERSATILE ROUTE TO IMPROVE THE TOUGHNESS IN SOFT MATERIALS

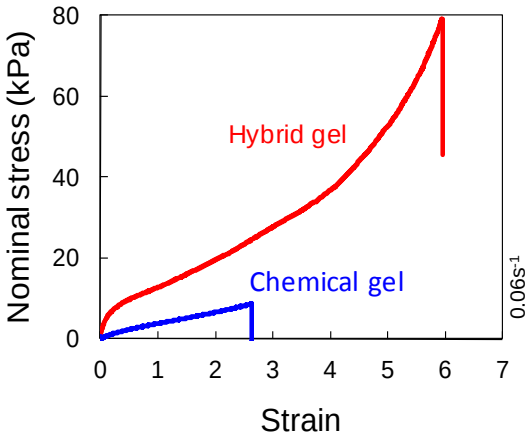
The idea of a tough hydrogel was once a paradox. Until recently, this class of soft materials was often regarded as weak and fragile due to their highly swollen structure. However, recent demand for tough polymer materials in biomedical applications has led to developments of original macromolecular architectures.



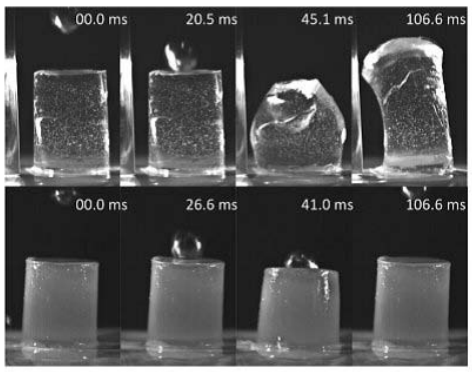
Our group developed new gels, combining the properties of associating polymers and those of covalent networks. Taking advantage of the strong and yet reversible adsorbing behavior of poly(N,N-dimethylacrylamide) chains (PDMA) with silica nano-particle surface, gels have been specially designed to obtain a well-controlled architecture with a homogeneous dispersion of spherical nano-particles into a weakly chemically cross-linked PDMA matrix. Silica content was varied, keeping the composition of the gel matrix fixed (an hydration at 88wt.%). Mechanical analysis of hybrid hydrogels in their preparation state points out a very unusual combination of properties induced by the incorporation of silica nano-particles into the chemical hydrogel: elasticity, dissipation, strength and strain at failure were seen to be enhanced simultaneously.

The strong physical interactions, taking place inside the gel, between PDMA chains and nano-silica particles, enable the network to accommodate reversibly its connectivity under stress. A rapid recovery of the network connectivity was seen to be achieved typically for time-scales of 1-10 seconds. The main feature of this work is the remarkable role played by silica nano-particles in the network to promote transient and recoverable connectivity. Such qualitative analysis of network dynamics opens up interesting prospects for toughening, self-recovering or self-healing applications.

The coupling strategy between covalent and physical interactions affords a very nice toolbox for controlling the structure/properties relationships of soft materials and open the way to new developments in biomedical technologies.



Tensile mechanical behavior at 0.06 s^{-1}: for the chemical gel (without silica) and the hybrid gel (containing 0.22 silica volume fraction).



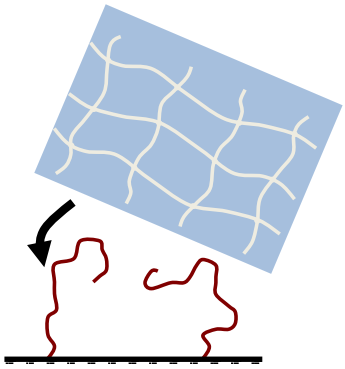
Chronophotographs demonstrate the falling of a 25 mm steel bead on hydrogels: It illustrates the impact of nano-particles on the mechanical behavior of the nano-hybrid hydrogels. Above, the sample without particles; compared to the highest content of particle (0.22 silica volume fraction).

C. Creton, D. Hourdet, A. Marcellan, T. Narita, S. Rose (PhD), L. Carlsson (M2), W. Fan (M2), M. Trouilh (M2), A. Dizeux (M2)

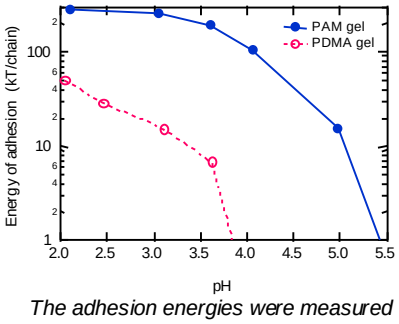
- Publications: [147,161,208], 4 invited conferences
- Support: ANR ADHGEL

UNDERWATER ADHESION: HOW TO TUNE MOLECULAR INTERACTIONS FOR SWITCHABLE ADHESION OF HYDROGELS

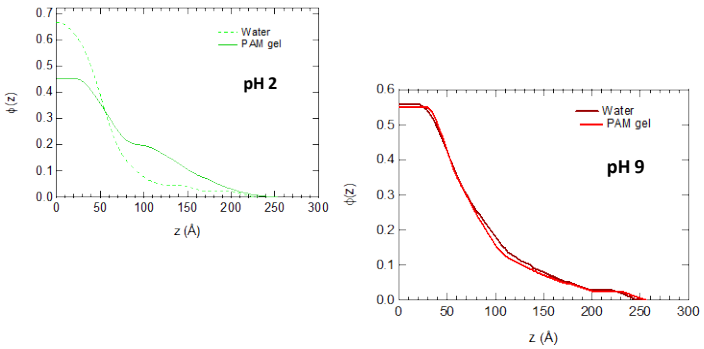
Hydrogels are of great interest in the biomedical field and as model systems for the study of living tissues. Since they contain large amounts of water, they are generally fragile and not naturally sticky. To understand the mechanisms that control the adhesion between hydrogels and surfaces, we have investigated simple model systems: Surfaces functionalized with polymers able to interact with hydrogel networks. The molecular interactions come from hydrogen bonds and can be triggered by a change in pH.



We selected chemically cross-linked neutral hydrogels of Polydimethyl acrylamide (PDMA) and polyacrylamide (PAM) which are weakly dissipative and insensitive to their environmental conditions. pH sensitive poly(acrylic acid) brushes were synthesized by either the "grafting onto" or the "grafting from" approach. The "grafting onto" strategy is straightforward and can be easily used with only small amounts of commercially available polymers. We were also able to synthesize poly(acrylic acid) brushes with the surface-initiated "grafting from" approach using controlled radical polymerizations (ATRP or RAFT).
The characterization of the interactions between the hydrogel and the brush was performed as a function of pH at two different scales: molecular and macroscopic. At the scale of the brush, we were able to determine the effect of the adsorption of the hydrogel on the conformation of the surface-attached chains using neutron reflectivity. Specific interactions, here hydrogen bonds, stretch the chains similarly to the electrostatic repulsion, indicating the interpenetration of the brush within the gel. The macroscopic method consisted of a novel underwater adhesion test based on contact mechanics and developed in the laboratory. The possibility to tune reversibly the adhesive properties of the gel with the pH was demonstrated by a stronger adhesion when specific interactions were present.



The adhesion energies were measured with a new home-made device based on flat punch tests in water. Working underwater removes the surface tension effects and allows a direct probe of specific interactions between the hydrogel and the brush. The surprising difference of adhesion energies found here for PAM and PDMA gels is indeed due to kinetic effects (kinetics of the formation of specific interactions and kinetics of disentanglement of chains).



The contact of a PAM hydrogel and PAA brush was probed at the molecular scale by using neutron reflectivity. The density profiles of the PAA brush in the presence of pure water and in contact with the gel were determined. The brush is stretched by the presence of the gel for pH = 2. The H-bond interactions between the brush and the gel have an effect as strong as that of electrostatic interactions on the stretching of the brush.

C. Creton, D. Hourdet, Y. Tran, E. Siband (PhD), G.Sudre (PhD), S. Sanjuan (PhD)

- Publications: [86,87,259,260,261,262,183,225], 8 invited conferences
- Support: ANR ADHGEL

Objectives for 2019

The target for 2019 is to consolidate the internal collaborations that we have developed, strengthen our collaborations with polymer chemistry groups and focus on truly interdisciplinary projects. The topic of pressure-sensitive-adhesives is now mature and new ideas will only come from innovative chemistry. However adhesion under water, applications of hydrogels as responsive valves and the relation between molecular and microstructural dynamics and macroscopic properties still holds great potential for further development. With the help of industry we plan to develop further our research on fracture mechanisms of soft materials. Possible new directions for 2019 could be the investigation of interfaces structure and mechanics where interfacial transport is important such as solvent permeation or electrical or ionic conduction.

Conclusion

This project focuses around the central theme of mechanical properties of soft polymer materials. Building on a unique combination of expertise ranging from the molecular level to the mesoscopic level the project holds promise to develop original investigation methods an insight on large strain properties of soft polymer based materials. The expansion of the last few years in size and visibility of this activity testifies of the correct choices that were made five years ago. Challenges for the future is to keep abreast of the competition and strike the right balance between new research topics and consolidation of existing ones.

Involved Researchers

L. Bokobza, B. Bresson, M. Ciccotti, C. Creton, G. Ducouret, D. Hourdet, A. Marcellan, T. Narita, Y. Tran

Scientific Production

29 Invited conferences in international meetings, 100 publications in refereed journals, 1 patent.

35% of the publications were on adhesion related topics, 27% on rubber related topics and 23% on hydrogels. These papers have been cited ~1000 times.

61 are in collaboration with other research institutions including 42 with foreign research institutions coming from 12 countries (USA, UK, Spain, Belgium, Germany, Netherlands, Japan, Greece, Australia, Czech Republic and India).

Resources

13 PhD students:
- CIFRE or Industrial Fellowships: A. Cristiano (DSM), S. Mzabi (Michelin), E. Ducrot (DSM), C. Wable (St-Gobain), J. Wollbret-Blitz (Michelin)
- ANR Funded Fellowships: G. Sudre (ADHGEL), J. de Crevoisier (AMUFISE),
- University Fellowships: S. Sanjuan (UPMC), G. Miquelard-Garnier (UPMC, J. Nase (UPMC), E. Siband (UPMC), S. Rose (UPMC)
- European Project Fellowships: F. Deplace (N-Shape), E. Degrandi (NAPOLEON), F. Tanguy (MODIFY).

21 Post-docs:
C. Carelli (NAPOLEON), N. Glassmaker (NAPOLEON), T. Yamaguchi (NAPOLEON), J. Marchal(NAPOLEON), L. Sonnenberg(NAPOLEON), C.J. Lin (ADHGEL), S. Nguyen (NAPOLEON), G. Hu (MECHYBRIDES, Urgo), O. Ramos (MODIFY), D. Martina (MODIFY), H. Zhang (AMUFISE), N. Wanakule

(Urgo), S. Bhuyan (MODIFY), C. Karakasyan (Saint-Gobain, Brothier, NANOSTIM), A. Kotzev (ATER), C Cannizzo (ATER), S. Lack (Brothier), M. Legros (Brothier), A-M. Layre (ANR NANOSTIM), Y. Kadam (CEFIPRA), H. Zhang (Michelin).

25 “Master 2” students and 22 Eleves Ingénieur.

3 European RTD projects (N-Shape MODIFY and NAPOLEON) on adhesion related topics for a total budget of 1,3 M€.
3 PhD Students funded by industry (Etienne Ducrot, Samy Mzabi, Clémence Wable) for total budget of 450 k€.
7 years of post-doc funded by industry (N. Wanakule, H. Zhang, C. Karakasyan 2 yrs, M. Legros, S. Lack, G. Hu).
4 ANR projects (Adhgel, Mechybrides, Nanostim, Amufise) for a total budget of 600 k€.
1 indo-french project CEFIPRA (Yogesh Kaddam) for a total budget of 150 k€.

TAILORING INTERFACES AND MESOSCOPIC STRUCTURES

Introduction

The macroscopic usage properties of complex heterogeneous systems such as suspensions, emulsions and foams are determined by the structure and interfacial properties of its constituents at the mesoscale. Dramatic practical examples are the flow properties of a cement paste that depend on the aggregation state of the mineral particles in the suspension, and foam stability that can be altered by surfactants conformation and dynamics. In general terms, our goal has thus been to identify, modify and control the mesoscopic chemical structure or the physical phenomenon impacting the behavior of soft formulated systems. However, the physical phenomena controlling the targeted properties are often hidden in complex formulated systems and cannot be revealed by parametric testing or numerical modeling as commonly performed in engineering departments. Oppositely, simple experiments used to validate physical concepts cannot capture the full range of behaviors expected from real systems. Our approach therefore had to be dual and this is why both physicists and chemists were involved in this project: we designed quite successfully controllable chemical formulations and as well as the physical experiments able to reveal the mesoscopic physical lever to the macroscopic physical and mechanical properties.

An originality of our project has been a particular focus on responsive macromolecular systems. Indeed, within our stated objective to impact macroscopic properties, chemistry offers an opportunity to force metastable mesoscopic structures and make them respond to external stimulus such as light or temperature.

Beyond this unity of interest and method, our research can be subdivided in three general areas: organomineral assembly (stability and rheology control by polymers), transfer (wetting and drying) and switchable interfaces (foam and emulsions).

State of the art and international context

This project is at the cross-road of different knowledges. Solid-state chemists know how to elaborate organized hybrid structures by a bottom-up approach. Soft Matter physicists, following the lead of de Gennes, have devised the scaling laws relevant to polymers thermodynamics in solutions at interfaces. There has been a lot of activity to transpose this knowledge in our field but the scale remains below the micrometer and the macroscopic scale can usually not be reached. At the other end of the spectrum, laboratories in engineering departments were able for instance to produce complex emulsions by microfluidic means or to devise a phenomenological description of the mechanical and transport properties of composite materials from composition and texture analysis. While this engineering approach has in the past allowed significant advances, in particular in the formulation of reinforced elastomers and high strength concretes, it is difficult to go further along this line as the formal understanding of the link between the chemistry and the macroscopic properties is sometimes lacking.

With our multi-scale approach, we combined the tool boxes of different disciplines: thermodynamics and energetic of colloids, polymer transfer in soft matter, hydrodynamics, as well as polymer chemistry. In this manner, we were able to control the properties of suspensions or foams by tailoring short and long range interactions. To achieve this, we put a large emphasis on the design of

smart core-shell nanoparticles and stimuli-responsive polymers. It can be said that we have an original and up-to-date mastering of the synthesis of polymers of well-controlled architecture or with the required stimuli-responsive properties. As a matter of fact, the development of home-synthesized macromolecules designed to stimulate interfaces and hence, to trigger macroscopic properties of dispersions (emulsions for instance) is now well established in our lab. Our approach was nevertheless not only bound to the creation of new molecules or colloidal objects but we also sought a deep understanding of the physical chemistry of the mechanisms involved at all the different scales that are responsible for the macroscopic changes. The mechanisms were studied so as to bridge the micro (deformation of polymer thin films, soap films instabilities, adsorbed polymer conformations)/macro (foam and suspensions stability, wetting contact angles, rheology) gap. To do so, we not only mobilized the relevant characterization techniques (spectroscopy, wave scattering, imaging, local and macroscopic mechanics) but also devised original to-the-point bench-top experiments (thin film pressure device, in-situ variable composition controlled pendant and spinning drop tensiometer, STRAFI measurement of molecular diffusion, ...).

International influence and attractiveness

Our reputation lies in our capability, while building on fundamental understanding, to actually provide the keys to solve real-life and intricate industrial problems. This, plus our multi-scale approach, places us at the cross-road of different disciplines and this makes us possible to secure numerous collaborations at the highest international level. It is significant to note that we are particularly attractive for international academic partners from Engineering departments (Wilson Poon, Edinburgh; Gerry Fuller, Stanford; Brad Chmelka, UCSB; Howard Stone, Princeton, Robert Flatt, ETH Zürich; N. Denkov, Sofia University) who are not able to reproduce in their home institutions our multi-disciplinary approach. Reciprocally, we are regularly invited in Engineering departments abroad (Crosby research group, UMass; Institut für Baustoffe, ETH Zürich). These collaborations are encouraged by industrial support which is a testimony of the fertility of our approach (Total, Sika, Schlumberger, Saint-Gobain, Givaudan, Nestlé, Rhodia…). The exemplarity of our approach has also been recognized at the European level since we are one of the three reference laboratories selected by the 7th European Framework Program to advise the Chemical Engineering Faculty of Sofia University in its capability built-up initiative (“BeyondEverest”).

Furthermore, our comprehensive approach and capabilities made us a partner of choice for regional (DIM Oxymore, Dim C’Nano), national (GIS Geopolymer, GdR Mousses et Emulsions, GdR Ornano) and international (GdR-I Multi-scale Materials Under the Nanoscope, Softcomp, Nanocem) research networks.

Insertion in the general orientations of the laboratory

Designing model systems and developing original instrumentation are two of the main tools deeply rooted in the general culture of our laboratory which were at the very basis of the project described in this section. Despite their clear academic orientation, these studies have received much attention from our industrial partners. The equilibrium which has been obtained between public and private funding has allowed a high level of student hiring and many international collaborations. In this sense also, this research is representative of the general approach of our laboratory.

Societal relevance

The control of soft interfaces is a fast moving current topic, which is of crucial importance in numerous domains of applications like medicine, pharmaceuticals, food industry, cosmetics, pesticides, construction or oil-based activities. As an example, our research on grafted gold particles

can be employed in various fields, particularly for biological applications (therapy by local hyperthermia). Several of our activities are directly related to sustainable growth issues such as: emulsions and encapsulation for storage and parsimonious delivery of active principles, Polymer dispersants for low CO_2 emission cements, Efficient mixing and dissolution for industrial processes.

Training of young researchers

Most of our former PhD students and Postdocs are now employed in the R&D departments of major industrial groups in the energy (Total, Schlumberger), food (Nestlé), building (Saint-Gobain) and chemistry (L'Oréal, Rhodia) sectors, a significant number of them outside of France. A minority of them nonetheless pursued a research career in academia (CNRS, universities)

Researchers involved in this project are major actors in two industrial Chairs (Saint-Gobain and Total) aiming at encouraging vocations for research in the fields of Materials, Surfaces and Soft Matter.

Most of our teaching duties are linked to our research activities. We are either responsible or involved in courses in colloids, polymer physical chemistry, and interfaces at the L3, and M1 level. At the M2 level, the UPMC and ESPCI faculty members neing part of this project are responsible for teaching units (UE) where our research results are directly transposed ("Formulation des dispersions colloïdales et des systèmes organisés", "Matériaux minéraux, minéraux industriels", "Rhéophysique des systèmes désordonnés").

Solved questions

Organo-mineral assemblies (Water-solid interfaces)

Cement superplasticizer, [116, 243]
Drawing on polymer physics concepts and on inorganic chemistry of oxides, we have shown how to control the rheological properties of cement pastes with high aluminum content by optimizing the structure of grafted polycarboxylates. Independently, we have designed a cement dispersing agent with sulfate resistance by partial substitution of carboxyl groups by trialkoxysilane in polycarboxylates.

Thermoresponsive aggregation of nanoparticles [154, 194]
We demonstrated that the aggregation temperature of gold nanoparticles can be easily modulated in a broad range of temperatures either by grafting modified commercial polymers or new synthesized polymers. The state of aggregation can be limited by forming hybrid polymer micelles.

Wetting and drying of complex fluids (Air-water-solid interfaces) [120, 128, 129, 184, 212, 228, 229]

We found that the dynamic wetting of polymer solutions is strongly slowed down in conditions where evaporation is fast: evaporation drives the formation of a very viscous zone over a few nanometers at the edge of the drops where polymer accumulates.

When a drop of water spreads on a polymer thin-film, the contact angle is controlled by the rate of transfer of water evaporating from the drop and swelling the substrate ahead of the contact line.

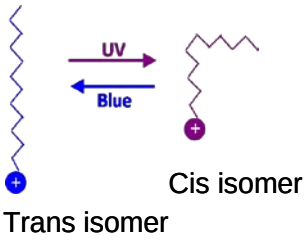
Photoswitchable interfaces (Fluid-fluid interfaces):

Emulsions [21, 177, 196, 197]
We have photocontroled emulsions stability through surfactant mesophases / photoresponsive polymers interactions.
Foams [179, 190, 240]

We have produced the first photoresponsive foams and emulsion foams using photoswitchable surfactants or polymers. We have shown that the surface tension is controlled by the light intensity through a change of the surfactant adsorption/desorption dynamics. Hydrodynamical instabilities can be nucleated in the thin-liquid films made from these surfactants through a change of molecular interactions.

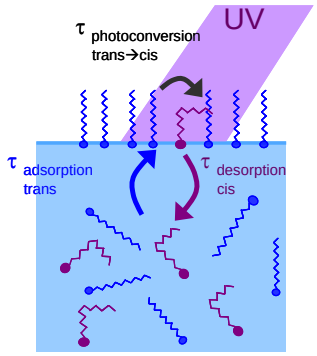
CONTROLLING LIQUID INTERFACES WITH LIGHT : FROM PHOTOSURFACTANTS TO PHOTOFOAMS

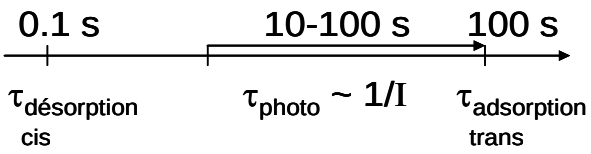
Our goal is to control interfacial dynamics using light stimulation. Light is easy to focus in space and time. We use a cationic photoswitchable AzoTAB, containing an azobenzene group on the hydrophobic tail, which switches from a cis to a trans conformation when the wavelength is switched from UV to blue. We follow the cascade of mechanisms from the molecular scale to the macroscopic scale generated by this stimulation..



Adsorption dynamics

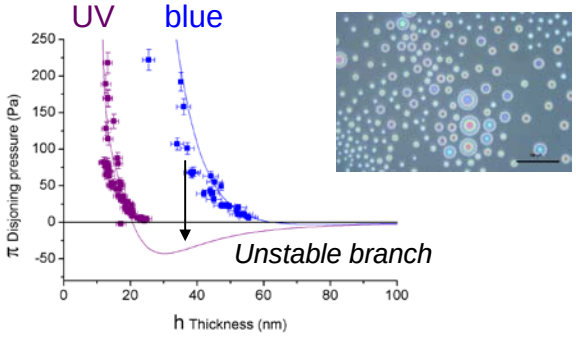
By investigating the adsorption dynamics of cis-trans mixtures we find that the desorption of the cis is very fast (0.1 sec) compared to the trans adsorption (100 sec). Hence when UV light is shone on the samples, adsorbed trans surfactants convert into cis which quickly desorb from the interface. The rate of photoconversion controls thus the surfactant concentration at the interface and therefore light intensity controls the surface tension!





Instabilities in thin-liquid films

Thin-liquid films are stabilized by these photosurfactants and stimulated with UV light. Surprisingly the films remain stable and various instabilities are nucleated in the films. We find that they are due to a change of the molecular interactions between the liquid interfaces of the films switching from a stable to an unstable branch of the disjoining pressure isotherms.

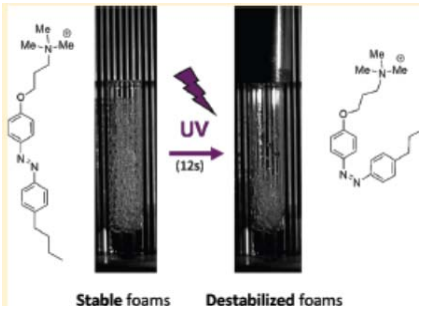


Left: Disjoining pressure of thin-liquid films stabilized with azoTAB with various cis/trans ratios.

Right: Spherical caps are nucleated in the films upon UV stimulation and observed using light interferometry.

Photofoams: remote control of foam stabilization using light

We find that a foam made from a trans-rich solution is stable but can be destabilized by shining UV on it. The foam stability depends on the light intensity and on the amount of cis/trans in the solution.



C. Monteux, F. Lequeux, C. Tribet, E. Chevallier (PhD), A. Mamane (M2)

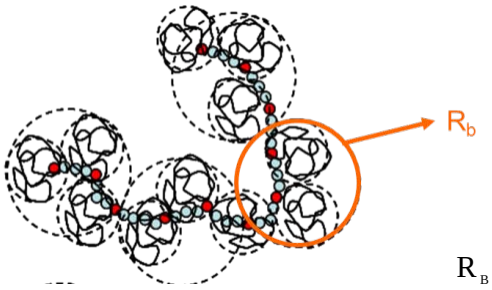
- Publications: [190,240, patent CNRS/UPMC FR 1151242 (2011)]
- Collaborations: H.A. Stone (Princeton), I. Cantat et A. Saint-Jalmes (IP Rennes)

ORGANO-MINERAL INTERACTION AND RHEOLOGICAL CONTROL OF CEMENT PASTE

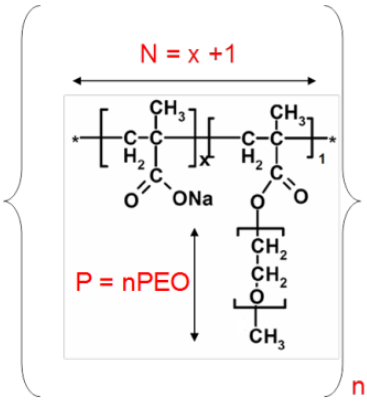
Cement polymer plasticizer

The fluidity of highly concentrated mineral pastes can be improved by adsorption of polymers on the mineral surfaces. In practice, the cement industry uses comb polyelectrolytes. Electrostatically adsorbing on the cement grain, they increase particles repulsion through steric effects and maintain the fluidity of the paste.

In solution, the polymer conformation can be predicted by scaling laws from its molecular architecture:

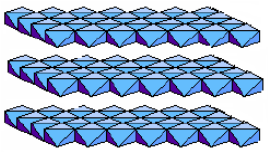


$$R_B = a \frac{a_P}{a_N} P^{\frac{7}{10}} (x+1)^{\frac{-1}{10}}$$



Cement aluminum hydrates

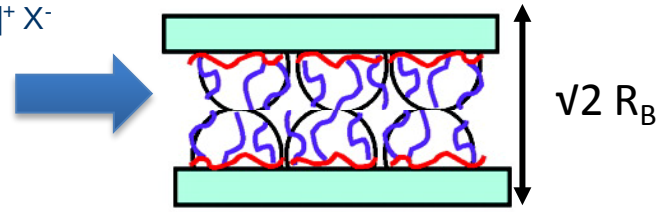
One of the result of cement hydration is a layered double (Ca, Al) hydroxyde. This lamellar phase is positively charged and intercalates anions.



$[Ca_2Al(OH)_6]^+ X^-$

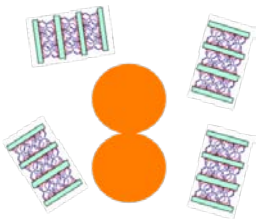
Sequestration of the plasticizer as an organo-aluminate phase

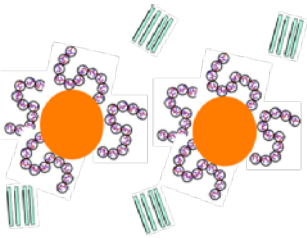
We have shown on model systems by NMR, SAXS and SANS that a stable intercalate forms from the polyelectrolyte and the aluminate. Its basal spacing and stability depends on the polymer structure and conformation.



How to prevent the sequestration of the plasticizer by the aluminate and maintain its dispersing capabilities?

Adapt its structure and its conformation to prevent the formation of the intercalated structure!

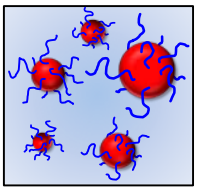




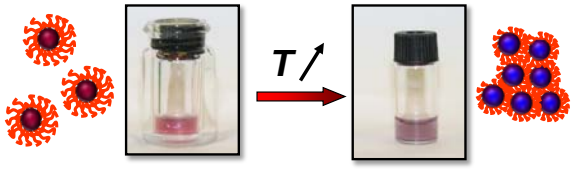
J.-B. d'Espinose, Z. Souguir (Post-doc), C. Lenain (PhD)
Publications: [116] (feature article)
Support: Sika
Collaborations: Université de Bourgogne (UMR 5209)

TUNABLE, CONTROLLED AND REVERSIBLE ASSEMBLIES OF GOLD NANOPARTICLES

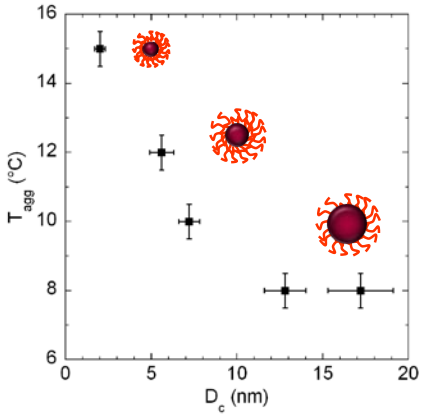
The ability to control reversibly the assembly of nanoparticles is a strong concern in many application domains, including biodiagnostics, drug delivery and their assembly in 1D, 2D, or 3D remains a challenge. To achieve this goal, one of the promising ways is the use of *stimuli* responsive polymers.



Modified and synthesized thermosensitive polymers were grafted on nanoparticle surface. In the case of polymer grafted gold nanoparticles, the **reversible aggregation** can be induced either by an increase or a decrease of the temperature, i.e. using polymers exhibiting a **LCST or UCST**.

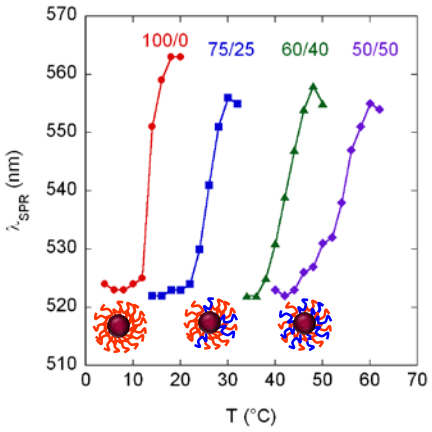


a

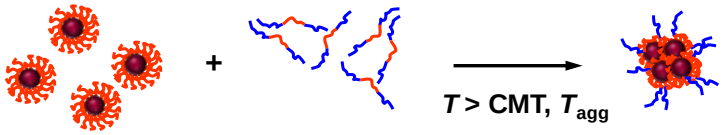


For LCST polymers**, the aggregation temperature of the polymer-grafted gold nanoparticles can be precisely modulated** in a broad range of temperature by changing either the **size of the nanoparticles** (figure a) or **using polymers with different hydrophobicity** (figure b).

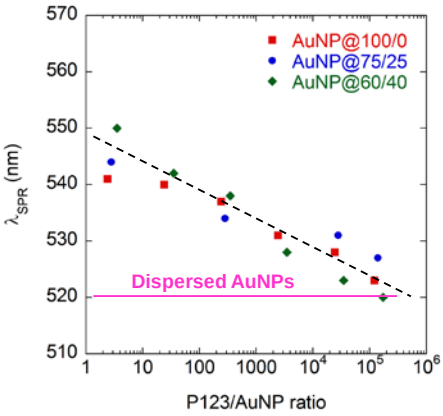
b



Aggregation state control



The aggregation can be controlled by adding in solution polymeric surfactants which allow controlling **the size of the aggregates** (nanoparticle number) and avoid macroscopic precipitation.



Applications

Both *stimuli*-responsive grafted gold nanoparticles, i.e. with LCST or UCST polymers, open great opportunities in biomedical applications such as **hyperthermia** or **biomolecules detection**.

N. Sanson, N. Lequeux, C. Durand-Gasselin (PhD)

- Publications: [154, 194]
- Collaborations: Bruno Palpant, Simona Laza (LPQM/ECP) , Cécile Sicard, Remita Hind (LPS/Paris Sud), Marie Carriere (LSDRM/CEA), Philippe Banet (LPPI/U. Cergy)

Objectives for 2019

Our research lead to significant advance in the design of systems for flow control, wetting of polymer thin films, and stabilization/destabilization of foams or emulsions. The emphasis was mainly on home-made molecular synthesis, interfaces composition and structures. Recently, we have investigated and modeled interfacial dynamics of stimuli-responsive systems. While keeping the same goal and methods, we now wish to extend our understanding of the role of interfacial dynamics to a wide variety of original systems. This will be an essential part of two of the new projects "Comprehensive engineering of pastes and powder" and "Engineering of liquid interfaces for encapsulation". For example, in the project "Comprehensive engineering of pastes and powder", to keep with systems we have studied in the past, we plan to study the dynamics of adsorption of polymers in relation to the surface accessibility to water. This point is crucial to mitigate the effect of dispersant on the retardation of cement hydration.

The expertise we have built-up on responsive interfaces will also be an essential part of the project "Engineering of liquid interfaces for encapsulation". This project requires the design of polymer molecules for the stabilization/destabilization of double emulsions, the practical development and thus commercialization of these products being presently impeded by the difficulty to obtain both responsive and stable emulsions. The achievement of new systems with tunable interfacial dynamics relies on the creation of original species adsorbed at interfaces. Consequently, our objectives will also focus on the synthesis of multistimuli-responsive (organic-inorganic hybrid) particles. Such an approach will give the opportunity to reinforce our expertise in the synthesis and design *à façon* of molecules active at interfaces. The "Engineering of liquid interfaces for encapsulation" project will also requires the development (instrumentation) of techniques and methods along the line of what we have already performed in order to probe the rheological and mechanical properties of the interface.

Conclusion

By a fundamental, comprehensive and multiscale approach, we have been able to unlock industrial engineering problems in seemingly diverse areas such as emulsion or foam photo-control, cement rheology, and polymer wetting. All the questions we addressed shared the common challenge of interplay of physics and chemistry at the mesoscale in soft multi-component systems. This interdisciplinary approach with chemical synthesis of new objects and original design of physical set-ups will be pursued in the future projects, especially in the project "Engineering of liquid interfaces for encapsulation". This project will be centered around the creation of new objects particles adsorbed at interfaces for capsule stabilization on the one hand, and interfacial mechanics and (stimulated) transfer characterization on the other hand.

Involved researchers

B. Bresson, J.-B. d'Espinose, F. Lequeux, N. Lequeux, C. Monteux, N. Pantoustier, P. Perrin, N. Sanson, L. Talini, C. Tribet, H. Van Damme, E. Verneuil.

Scientific output

76 publications in peer-reviewed journals
9 invited conferences
1 patent

Resources

9 Postdocs:
A. Bourlon, C. Lenain, and C.N. Chao (3 month-contract Nanocem), G. Guena and J. Wang (Geoservices), Z. Souguir (CNRS), G. d'Espinoza (ANR), E. Rotureau (ATER UPMC), S. Piogé (ATER UPMC)

10 PhD students:
- CIFRE fellowships : A. Bourlon (IF PEN), C. Lenain (ATILH-SIKA), A. Tay (Rhodia), S. le Tirilly (Givaudan), S. Moro and M. Van Landeghem (Saint-Gobain), J. Dupas (Nestlé), W. Fan (Italcimenti)
- UPMC fellowships : E. Chevallier, A. Roudot, C. Durand-Gasselin

37 Masters and undergraduate internships:
- 18 Elèves Ingénieur,
- 9 Licence, Licence Pro, Master Pro
- 10 Master 2

Grants and ANR contracts:
3 ANR : Puit CO2, Depsec, PhiREM
SESAME Ile de France (equipment),
France-Stanford award (travel grant)

European Networks:
Soft Comp (European network of excellence, travel grants, experiments),
Beyond Everest (European research network, travel grants),
Nanocem (Industrial european research consortium, short term postdoc salaries, research equipment)

Industrial contracts:
Rhodia (1 CIFRE), Saint-Gobain (3 CIFRE, 4 short contracts), Nestle (1 CIFRE), IFP (1 CIFRE, 2 contracts), ATILH (1 CIFRE), Schlumberger (2 short contracts), Givaudan (1 CIFRE), Geoservices (2 postdocs), Lafarge (1 short contract), Bouygues (1 contract)

ANNEX: THE MECHANICAL/RHEOLOGICAL PLATFORM

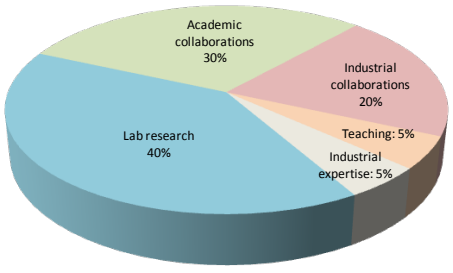
The mechanical platform is placed under the responsability of UMR 7615 (G. Ducouret) and linked to two research institutes within University UMPC : Institut des Matériaux de Paris Centre (FR2482) and the Fédération Dynamique des Systèmes Complexes Hors Equilibre (FED 21).

The diversity of our equipment allows the characterization of a very wide range of materials going from very fluid formulations to the very stiff materials by way of the soft gels, yield stress fluids, more or less concentrated, colloidal suspensions, pastes, granular materials as well as elastomers.

The plateform is equipped with conventional devices and tools developed specifically in the framework of new research activities, including the coupling macro–microrheology in association with Tetsuaru Narita. In the same way, the Surface Fluctuations Specular Reflexion spectroscopy (SFSR), a non-conventional technique developed in the lab, for high-bandwidth analysis of liquids and soft viscoelastic solids is available in the platform.

Conventional devices	*Non-conventional tools*
1 capillary viscometer	Surface fluctuations (SFSR)
5 controlled stress rheometers	Diffusing wave spectroscopy (DWS)
3 controlled strain rheometers	
4 tensile devices	
1 dynamic mechanical analysers	

Approximately 40 % of the activity is dedicated to the internal research of the laboratory. The mechanical platform is largely open to the socio-economic environment and the academic local and national communities. The activity is shared according to the scheme below:



SCIENTIFIC OUTPUT

Publications

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253. Papon, A., Montes, H., Hanafi, M., Lequeux, F., Guy, L. and Saalwachter, K., Glass-Transition Temperature Gradient in Nanocomposites: Evidence from Nuclear Magnetic Resonance and Differential Scanning Calorimetry. *Physical Review Letters*, 2012. **108**(6).
254. Papon, A., Montes, H., Lequeux, F., Oberdisse, J., Saalwachter, K. and Guy, L., Solid particles in an elastomer matrix: impact of colloid dispersion and polymer mobility modification on the mechanical properties. *Soft Matter*, 2012. **8**(15): p. 4090-96.
255. Pellizzi, E., Lattuati-Derieux, A., d'Espinose de Lacaillerie, J.-B., Lavédrine, B. and Cheradame, H., Reinforcement properties of 3-aminopropylmethyldiethoxysilane and N-(2-Aminoethyl)-3-aminopropylmethyldimethoxysilane on polyurethane ester foam. *Polymer Degradation and Stability*, 2012(in press).
256. Raudsepp, A., Fretigny, C., Lequeux, F. and Talini, L., Two beam surface fluctuation specular reflection spectroscopy. *Review of Scientific Instrument*, 2012. **83**.
257. Sanson, N., Bouyer, F., Destarac, M., In, M. and Gerardin, C., Hybrid Polyion Complex Micelles Formed from Double Hydrophilic Block Copolymers and Multivalent Metal Ions: Size Control and Nanostructure. *Langmuir*, 2012. **28**: p. 3773-82.
258. Souguir, H., Ronsin, O., Larreta-Garde, V., Narita, T., Caroli, C. and Baumberger, T., Chemo-osmotically driven inhomogeneity growth during the enzymatic gelation of gelatin. *Soft Matter*, 2012. **8**(12): p. 3363-73.
259. Sudre, G., Hourdet, D., Cousin, F., Creton, C. and Tran, Y., Structure of surfaces and interfaces of poly(N,N-dimethylacrylamide) hydrogels. *Langmuir*, 2012. **28**: p. 12282-87.
260. Sudre, G., Olanier, L., Tran, Y., Hourdet, D. and Creton, C., Reversible adhesion between a hydrogel and a polymer brush. *Soft Matter*, 2012. **8**: p. 8184-93.
261. Sudre, G., Siband, E., Hourdet, D., Creton, C., Cousin, F. and Tran, Y., Synthesis and Characterization of Poly(acrylic acid) Brushes: "Grafting-Onto" Route. *Macromolecular Chemistry and Physics*, 2012. **213**(3): p. 293-300.
262. Sudre, G., Tran, Y., Creton, C. and Hourdet, D., pH/Temperature control of interpolymer complexation between poly(acrylic acid) and weak polybases in aqueous solutions. *Polymer*, 2012. **53**(2): p. 379-85.
263. Wintgens, V., Layre, A.M., Hourdet, D. and Amiel, C., Cyclodextrin Polymer Nanoassemblies: Strategies for Stability Improvement. *Biomacromolecules*, 2012. **13**(2): p. 528-34.
264. Yan, X.L., Kitahama, Y., Sato, H., Suzuki, T., Han, X.X., Itoh, T., Bokobza, L. and Ozaki, Y., Laser heating effect on Raman spectra of styrene-butadiene rubber/multiwalled carbon nanotube nanocomposites. *Chemical Physics Letters*, 2012. **523**: p. 87-91.
265. Zhang, H., Scholz, A.K., de Crevoisier, J., Vion-Loisel, F., Besnard, G., Hexemer, A., Brown, H.R., Kramer, E.J. and Creton, C., Nanocavitation in Carbon Black Filled Styrene-Butadine Rubber under Tension Detected by Real Time Small Angle X-ray Scattering. *Macromolecules*, 2012. **45**(3): p. 1529-43.

Patents

B1. INPI 0908650, 2009, Panneaux de construction en plâtre. Extension PCT WO 133886. BPB Ltd, Saint-Gobain, R. Fisher, H. Van Damme, R. Walton.

B2. INPI 1054969, 2010, Procédé de réalisation d'une couche mince de silicium monocristallin sur une couche de polymère. Extension PCT WO 161122. CEA, CNRS, M. Argoud, H. Moriceau, C. Fretigny.

B3. INPI 1151242, 2011, Tensioactifs photostimulables. Extension PCT 50327. CNRS, UPMC, C. Monteux, E. Chevallier, F. Lequeux, C. Tribet.

B4. INPI 1251722, 2012, Microcanal avec dispositif d'ouverture et/ou fermeture et/ou pompage. ESPCI, CNRS, Y. Tran, C. Vergne, L. Devys, F. Monti, P. Tabeling.

Invited conferences

International conferences

Drying of mutlicomponent latex suspensions.
Secret of formulation by the SCI, London, July 2007
F. Lequeux

Optimizing the Structure of a Soft Material for Adhesive Properties.
2nd LSW Symposium on Soft and Wet Matter, Sapporo, Japan, 2007
C. Creton

Responsive interfaces grafted with polyelectrolyte or polyampholyte.
SPIE Smart Materials, Nano- & Micro-Smart Systems, Adelaide, Australia, 2007
Y. Tran

Adhesion Promotion onto Polyolefin Substrates.
AWA Self-Adhesive Seminar, Amsterdam, 2007
C. Creton

How can tensile tests be useful to design optimized PSA?
TECH 30 Global Conference VI, PSTC 2007, Orlando, USA, 2007
C. Creton and F. Deplace.

Tack between immiscible uncrosslinked elastomers.
ACS 171st Spring Technical Meeting of the Rubber Division, Akron, USA, 2007
R. Schach *et al.*

Lateral Heat diffusion in Layered Structures: Theory and Photo-Thermal Experiments. *International conference on Photoacoustic and Photothermal Phenomena*, Le Caire, Egypte, 2007
D. Fournier and C. Fretigny

Linear and non-linear mechanics of filled elastomers, gradient of glass transition temperature and geometrical effects.
Reinforced Elastomers: Fracture Mechanics, Statistical Physics and Numerical Simulations,EUROMECH Colloquium 502, Dresde, 2008
F. Lequeux

Strategies to improve adhesion of waterborne acrylic PSA on polyethylene.
Technical Seminar FINAT 2008, Barcelona, Spain, 2008
C. Creton

Strategies to Enhance Adhesion on Polyolefin Surfaces.

AFERA Technical Seminar, Brussels, Belgium, 2008
C. Creton

Adhesion, friction and deformations at a nanometric scale on polymeric systems.
Seeing at the Nanoscale VI, Berlin, Germany, 2008
C. Fretigny

Pasty fluids modeling : a physicist point of view.
Ecole des Ponts - Peking University joint workshop Rheology of complex fluids: modeling and numerics, Paris, 2009
F. Lequeux

Wetting in the presence of drying.
4th LSW symposium on Soft and Wet Matter, Hokkaido University, 2009
F. Lequeux

What are the relevant lengthscales in platsicity of polymers.
14th International Conference on Deformation, Yield and Fracture of Polymers, Kerkrade, The Netherlands, 2009
F. Lequeux

From Local Polymer Dynamics to Mechanical Properties in Model Elastomer Filled with Nanoparticles.
Aquitaine Conference on Polymers, Arcachon, 2009
F. Lequeux

Fracture mechanisms of hybrid polymer gels in water
Gel Sympo 2009, Osaka, Japan, 2009
C. Creton *et al.*

Cavitation by Fracture of Model Rubber Networks.
Annual Conference of of the Korean Rubber Society, Jeju Island, Korea, 2009
A. Cristiano, A. Marcellan and C. Creton.

Intercalation of poly(methacrylate-gPEO) in LDH structures.
1st Sino-French Bilateral Seminar on Macromolecules and Soft Matter, Shangaï, Chine, 2009
J.-B. d'Espinose

Lateral heat spreading in layered samples.
MRS meeting, San Francisco, USA, 2009
C. Fretigny *et al.*

Friction and adhesion of rubber: let's twist again.
IOP Meeting, Londres, UK, 2009
A. Chateauminois and C. Fretigny

Surface tension effects in thin polymer films.
Confinement at the Nano-Scale in Polymers: Theoretical Predictions and Experiments, Poitiers, 2009
C. Fretigny

How filled elastomer mechanical properties are controlled by the glassy dynamics around the nanoparticles?
Polymeric Materials 2010, Halle (Saale), Germany, 2010
F. Lequeux

How does water wet hydrosoluble coatings?
2nd Sino-French Bilateral Seminar on Macromolecules and Soft Matter, Paris, 2010
F. Lequeux

Wetting in the presence of evaporation and solutes : where is the contact line?
Capillary Shaping of Solutes, Leiden, Netherlands, 2010
F. Lequeux

Nanocomposites: Why are they so interesting?
DPI Workshop on nanocomposites, Eindhoven, The Netherlands, 2010
C. Creton

Adhesion and Fracture of Soft Polymer Networks.
Gordon Conference : Polymer Physics, Mount Hadley, MA, USA, 2010
C. Creton

Advances in the Fracture Mechanisms of Filled Elastomers.
Macro 2010, Glasgow, UK, 2010
C. Creton

Mechanical Properties of Soft Viscoelastic Nanocomposites.
Macro 2010, Glasgow, UK, 2010
C. Creton

Tack and Adhesion of Soft Polymer Layers.
Workshop Dynasoft 2010, Cargese, 2010
C. Creton

Adhesion in water: Molecular interactions and macroscopic adhesion of hydrogels.
4th World Congress on Adhesion and Related Phenomena, Arcachon, 2010
C. Creton

Design of polyurethane-acrylic pressure sensitive-adhesives.
PSTC TECH 33 2010, Las Vegas, USA, 2010
C. Creton and F. Deplace

Polymer brushes: responsive and adaptative properties.
2nd Sino-French Workshop on Macromolecules and Soft Matter, Paris, 2010
Y. Tran

Adhesion and fracture of soft complex polymer networks.
2nd International Conference on Polymer Processing and Characterization, Kottayam, India, 2010
C. Creton *et al.*

Quantitative and semi-quantitative mechanical measurements at a submicrometric scale.
Nanobrücken : Nanomechanical Testing Workshop, Saarbrück, Germany, 2010
C. Fretigny

Heat diffusion in layered samples: 3w and thermoreflectance methods.
International Workshop on Photoacoustic & Photothermal Phenomena, Erice, Italia, 2010
D. Fournier, J.-Y. Duquesne and C. Fretigny

How filled elastomer mechanical properties are controlled by the polymer dynamics around the nanoparticles?
KIPS-ESPCI Workshop on Polymer Science 2011, Kyoto, 2011
F. Lequeux

Tunable, controlled and reversible assemblies of thermosensitive polymers grafted nanoparticles.
KIPS-ESPCI Workshop on Polymer Science 2011, Kyoto, 2011
N. Sanson

Adhesion in Water: molecular interactions and macroscopic adhesion of hydrogels.
3rd French-Brazilian meeting on Polymers, Florianopolis, Brazil, 2011
C. Creton

Toward understanding toughness of soft polymer materials.
Dutch Polymer Institute Annual Meeting, Zeist, the Netherlands, 2011
C. Creton

Sticky adhesives: from polymer physics to mechanics of soft materials .
11th triennial conference on the Science and technology of Adhesion and Adhesives, York, UK, 2011
C. Creton

Fracture of rubbers and hydrogels: from bond breakage to fracture toughness.
Seminar for the 80th anniversary of HH Kausch, Lausanne, Switzerland, 2011
C. Creton

Adhesion in water: molecular interactions and macroscopic adhesion of hydrogels.
60th conference of the Society of Polymer Science Japan, Osaka, Japan, 2011
G. Sudre *et al*.

Adhesion of hydrogels under water.
Gordon Conference on Adhesion Science, 2011. Lewiston, ME, USA
G. Sudre *et al*.

Nanoscale Investigation of the Condensation and Diffusion of Water at Crack Tips in Glass.
Material Science & Technology, Columbus, Ohio, USA, 2011
L. Ponson, G. Pallares, M. Georges, M. Ciccotti, E. Bouchaud.

Micro and Nanoscale Mechanisms of Subcritical Crack Propagation in Glasses.
Fractography of Glasses and Ceramics VI, Jacksonville, Florida, USA, 2011
M. Ciccotti, G. Pallares, S. Roux, L. Ponson, M. George

Multi-scale investigation of sub-critical crack propagation mechanisms in oxide glasses.
CECAM Workshop: Brittle Fracture at the Atomic Scale, Lausanne, Switzerland, 2011
M. Ciccotti, G. Pallares, S. Roux, L. Ponson, M. George.

Stick-slip dynamics in the peeling of an adhesive tape.
AFERA Technical Seminar 2011, Bruxelles, Belgia, 2011
M. Ciccotti

Polymer versus mineral glass fracture Space, time and energy dissipation scales.
Fracture and Flow of Advanced Glasses 5, St-Malo, France, 2011
M. Ciccotti.

Friction and adhesion within contact interfaces with rubbers: insights into local dynamics from field measurement.
Gordon Conference on Adhesion, Bates College, USA, 2011
A. Chateauminois *et al*.

Lateral versus perpendicular heat diffusion in layered structures: theory and experiments.
16th International Conference on Photoaccoustic and Photothermal Phenomena, Merida, Mexique, 2011
D. Fournier, J.-Y. Duquesne, C. Fretigny

Filled elastomer mechanics and polymer dynamics modification near surfaces.
IUPAC World Polymer Congress, Blacksburg, USA, 2012
F. Lequeux

Adhesion of hydrogels under water by hydrogen bonding: from molecular interactions to macroscopic adhesion.
American Physical Society March Meeting, Boston, USA, 2012
C. Creton

Viscoelastic adhesives: stickiness between the fluid and the solid.
1st International Conference on Biological and Biomimetic Adhesives, Lisbon, Portugal, 2012
C. Creton

Local friction of rubbers with rough surfaces.
15th International Conference on Deformation, Yield and Fracture of Polymers, Kerkrade, The Netherlands, 2012
A. Chateauminois

Nanocavitation in carbon black filled styrene-butadiene rubber probed by real timesmall angle X-ray scattering (SAXS).
International Rubber Conference 2012, Jeju Island, Korea, 2012
H. Zhang *et al*.

Nanocavitation in Nanocomposites Probed by Real Time Small Angle X-ray Scattering (SAXS).
IUPAC Macro 2012, Blacksburg, USA, 2012
H. Zhang *et al*.

What is geopolymerisation?

SoftCem 2012, International workshop on the microstructure, Setting and aging of cements: from soft matter physics to sustainable materials, Monte Verita, Ascona, Switzerland, 2012
J.-B. d'Espinose

National conferences

Propriétés mécaniques de films polymères confinés dans des contacts : viscoélasticité et plasticité.
Colloque National MECAMAT, Aussois, 2007
E. Gacoin, C. Fretigny, A. Chateauminois

Tension, énergie, (visco-)élasticité et contraintes de surface : solides et liquides.
GDR DynInter, Tour, 2008
C. Fretigny

Frottement et adhésion d'élastomères dans des contacts en torsion.
Congrès Français de Mécanique (CFM'09), Marseille, 2009
A. Chateauminois , C. Fretigny

Quelques expériences de rhéologie à une échelle submicrométrique.
Groupe Français de Rhéologie GFR44, Strasbourg, 2009
C. Fretigny

De nouveaux matériaux pour éradiquer les émissions de GES dans la construction.
Advancity, la ville post-carbone, Marne la Vallée, 2009
J.-B. d'Espinose

Nanoassemblages hybrides.
Rencontres Lafarge - CNRS, Paris, 2010
J.-B. d'Espinose

Viscosification et d'hydratation à une ligne de contact.
Journées de la matière condensée de la SFP, mini-colloque « liquides à l'échelle nanométrique », Montpellier, 2012
C. Monteux

Mesures mécaniques par AFM, application aux polymères.
Colloque National MECAMAT, Aussois, 2010
C. Fretigny

Les frottements du caoutchouc: stiction et stick-slip.
16ème Journées d'étude sur l'adhésion, Trégastel, 2011
A. Chateauminois *et al.*

Dynamique et rheologie d'interfaces liquides complexes.
J*ournées de formulation de la SFC*, Conférence-atelier sur la caractérisation des dynamiques aux interfaces liquides, 2012
C. Monteux

Prizes, awards

Dédale Prize 2007 of the French Adhesion Society, C. Creton.

Wake Medal 2011 of the UK Society of Adhesion and Adhesives, C.Creton.

Prize of the "Fondation Langlois" (2009), C. Monteux.

"Soft matter" Prize during the 8th Liquid Matter Conference 2011 (6-10th September 2011), Universität Wien in Wien, Austria, A. Papon

Lavoisier Prize 2008 de l'Apprentissage de l'UIC – Union des Industries Chimique Ile-de-France for the couple "Maître d'apprentissage - Pedagogical advisor", Y. Tran.

Prize of the best paper for 2007 in "Journal of Polymer Science B : Polymer Physics, F. Sosson *et al*.

"Highlight" in *Europhysics News* 39 (6) 2008 for a paper by A. Chateauminois and C. Fretigny in European Physical Journal E.

"Feature article" in *Journal of the American ceramics Society* (92 (11) 2009) for a paper by J.-B. d'Espinose

"Hot topic" in *Soft Matter* for a publication by A. Salonen, D. Langevin, P. Perrin, Soft Matter, 2010, 6, 5308-5311.

"Hot topic" in *Soft Matter* for a publication by D. Leiske, C. Monteux, N. Senchyna, H. Ketelson and G.G. Fuller, Soft Matter, 2011, 7, 7747-7753.

"Hot Topics" in *Soft Matter* for a publication by A. Papon, H. Montes, F. Lequeux, J. Oberdisse, K. Saalwachter and L. Guy. (2012)

Poster Prize of the 18th Oswald Colloquium (16th-18th May 2011), J. Dupas.

Poster prize at 15th International Conference " Deformation, Yield and Fracture of Polymers", DYFP2012. Y. Nziakou (Étudiant ESPCI, stagiaire) Rolduc (The Netherlands).

Best oral presentation in the topic Chemical Physics at the XLVIII International Students Scientific Conference, M. van Landeghem (10-14 april 2010) Russia.

Carl E. Carlson Prize for the best oral presentation at the international conference Polychar 19, Kathmandu, Népal. S. Rose.

Lectures in workshops

Plasticity, what we do understand and what we do not. Flow in Glassy System, Les Houches, 2007 F. Lequeux

Wetting and drying. Soft Interfaces, Les Houches, 2012 F. Lequeux

Stiction of adhesive contacts. Theoretical modeling and experimental simulation, Ecole thématique, Cargese, 2010 A. Chateauminois

Plasticity of amorphous polymer. Physics of Amorphous Solids: Mechanical Properties and Plasticity, Les Houches, 2010, F. Lequeux

Guest lecturer invited by the MRSEC program at U. Mass (host Al Crosby, Department of Polymer Science and Engineering) C. Monteux

Structure des hydrates cimentaires. 2nd Ecoles thématiques CNRS – ATILH « Matériaux Cimentaires », La Colle sur Loup, 2008J.B. d'Espinose

Structure des hydrates cimentaires. 3rd Ecoles thématiques CNRS – ATILH «Physique, Chimie et Mécanique des Matériaux Cimentaires», La Grande Motte, 2011J.B. d'Espinose.

Consultancy

Membership of scientific boards (Tarkett, Eurotab, Saint-Gobain and Solvay-Rhodia), (92 days) F. Lequeux.

71 days for 14 firms : 3M, Minneapolis, USA (10). 3M, Neuss, Allemagne (1). Cray-Valley, Creil (4).DSM Research, Geleen, Pays-Bas (14). Essilor, St-Maur (3). Faurecia, Stuttgart, Allemagne : (5). Henckel, USA (4). L'Oréal, Chevilly-Larue (22). Michelin, Clermont-Ferrand : (1). National Starch, Londres, Royaume-Uni (1). Saint-Gobain, Aubervilliers (1). Tesa ; Allemagne (1). Tetrapak : Suède (1). Wrigley, Chicago, USA : (3), C. Creton.

15 days for Hutchinson, Snecma, Eurocopter, PSA, A. Chateauminois.

Kermel, Colmar (2007-2012), AEC RTS, Troyes (2007-2009), Henri Selmer, Paris (2007-2012), J.L. Halary

Popular scientific works, miscellaneous

- Point de vue sur la dynamique des polymères en milieu confiné dans « La Recherche », Janvier 2009, C. Fretigny
- Ecriture de pages Wikipedia sur les mousses et surfactants, C. Monteux

- Montage de deux expériences grand public à l'Espace Pierre-Gilles de Gennes. Mars-Ocotbre 2008, C. Creton
- Tournage d'un petit clip avec la journaliste E. Lachaud pour une émission de E=M6 sur les combinaisons de natation. 12 Décembre 2008, C. Creton
- Tournage d'une vidéo pour la "Nuit des Chercheurs" sur le thème "Imaginez le futur". 28 septembre 2012, Y. Tran
- Tournage d'une séquence pour l'émission "On n'est pas que des cobayes" de France 5 sur le thème "Peut-on coller James au plafond?". Octobre 2012, E. Ducrot
- Conférences sur le thème« Pourquoi ça colle : du miel au post-it » pour le grand public à La Seyne les Alpes, aux journées "Paris de Sciences", au théâtre en région lyonnaise et pour les enseignants du primaire à l'IUFM de Rouen. 2007, 2008, 2009. C. Creton
- De la macromolécule au matériau polymère.
Union des professeurs de Physique et de Chimie, PariS de ScienceS, 2007.J.L. Halary
- La transition vitreuse des polymères thermoplastiques, élastomères et thermodurcissables : relation avec la structure macromoléculaire.
Forum Technologique TA Instruments, ENSAM Paris, 4 juin 2009
Colloque de Formation Apollor: le PET dans tous ses états.
Ecole des Mines de Nancy, 2009
JL Halary
- La physico-chimie au service de la formulation de matrices époxy à résistance au choc améliorée.
Workshop EADS, « Structure-propriétés des composites »Suresnes, 2010. J.L. Halary
- Rheology and Thermal Analysis of Polymers.
Journée sientifique internationale TA Instruments, ECPM Strasbourg, 2011. JL Halary
- Eléments de compréhension moléculaire de la déformation des matériaux polymères.
ENS Cachan, LMT, 2012. J.L. Halary
- Les pneus de voitures : comment la dynamique locale des polymères contrôle leur consommation d'énergie.
Formation permanente Professeurs de classes préparation X-ESPCI, 2012 Ecole Polytechnique 2h. F. Lequeux

Organization of conferences and editorial tasks

Organisation de 14th International Conference on Organized Molecular Films (ICOMF 14), 2012, Paris, P. Perrin, Y. Tran.
Organisation of mini-colloque « Systèmes auto-assemblés sous influence », Journées de la Matière Condensée (JMC 11), 2008, Strasbourg, Y. Tran.
Journées Jeunes Rhéologues, 2012, Saint-Rémy-les-Chevreuse, Groupe Français de Rhéologie, G. Ducouret.
Organisation of the Workshop 'particules aux interfaces', ESPCI, 2011, C. Monteux.
Organisation of mini-colloques on friction at JMC 11 (Strasbourg, 2008) and JMC 13 (Montpellier, 2012), A. Chateauminois.
Organisation of the workshop on Multi-Scale Dynamics of Structured Polymeric Materials, Paris, 2010, C. Creton.
Scientific and Organization committee of the 2nd sino-french workshop on Macromolecules and Soft Matter, Paris, 2010, C. Creton.
Program chair of the conference « Deformation, Yield and Fracture of Polymers », 2009. C. Creton.
Scientific and Organization committee of WCARP 4 in Arcachon, France, 2010, C. Creton.
Program chair of the International ESPCI/MICHELIN colloquium, 2011, F. Lequeux
Scientific committee of the "Journées Maghreb-Europe" MADICA 2008, 2012, C. Fretigny
Scientific committee of the research school Theoretical modeling and experimental simulation in tribology, 2010, C. Fretigny
Editorial board of *Tribology International*, A. Chateauminois
Editorial board of *Langmuir,* C. Creton
Editorial board of *Journal of Adhesion*, C. Creton
Editorial board of Statistical Mechanics: Theory and Experiments, F. Lequeux
Advisorial Board of *Applied rheology*, F. Lequeux
Guest editor of the special issue on biomimetic adhesion of the *MRS Bulletin*, May 2007, C. Creton
Guest editor of a special issue of *J. Polym. Sci. Polym. Phys*, C. Creton

Continuing education

Rheology platform : « Rhéologie pratique » Formation Permanente UPMC », 8 days, 2009, 2011, G. Ducouret
Adhésion : usage et choix pour vos applications industrielles, Collège de Polytechnique, 2012, Formation de 14h dont 3.5h sur "Physique et mécanique de l'adhésion" (M. Ciccotti) et 3.5h sur les "Aspects chimiques du collage" (C. Creton).
Cours sur les propriétés d'adhésifs en émulsion dans la formation « Principles of emulsion polymerization » organisée par POLYMAT, 2012, C. Creton.
Formation continue aux journées d'étude de l'adhésion, 2007-12, C. Creton.
Relations structure-propriétés dans les matériaux polymères, Cycle de conférences de formation de 24, Centre de Recherche Public Henri Tudor, Esch-sur-Alzette, Luxembourg, 4 journées, 2010.