GDR International
Mechanics of Nano-Objects
"Mecano"
Proposal 2016-2019

GDR CNRS mecano

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I. Context :

1.1. History

The "Mecano" (standing for "Mechanics of Nano Objects") International GDR has been supported by the CNRS for the past 4 years, from 2012 to 2015. This essentially European network included 31 French partners (including 4 industrial ones) and 11 laboratories from Germany, Austria, Italy, Switzerland, UK and Ireland. Before that, "Mecano" was a national GDR, (GDR CNRS 3180 MECANO, 2008-2011) involving 44 laboratories. These networks were lead by Olivier Thomas (IM2NP, Marseille) and co-workers (Anne Ponchet, CEMES-CNRS and Thomas Pardoen, Univ. Louvain) who are now leaving their front role in a very active community interested in the mechanical properties of small objects. The adjective "small" is purposely loosely defined here as the European expansion fueled a renewal of the topics of interest, encouraged by recent findings, new techniques and remnant fundamental questions. The common ground on which all the partners are working are condensed matter objects that posses at least one internal or external dimension smaller than a micron. Size effects can appear on various properties due to this structural confinement and the mechanical ones are at the core of the Mecano network.

1.2. Genesis of the current proposal

The last 4 years of Mecano have been extremely active (see the report by O. Thomas) and most of the participants returned positive feedback and expressed the will to continue. In agreement with the scientific committee O. Thomas suggested me as a possible successor. Following this, a new poll has been sent to the current GDRi participants this spring. It was aimed at defining the topics on which this proposal will be established. Three questions were asked about the possible topics, methods and objects. The results are presented in the following figures.

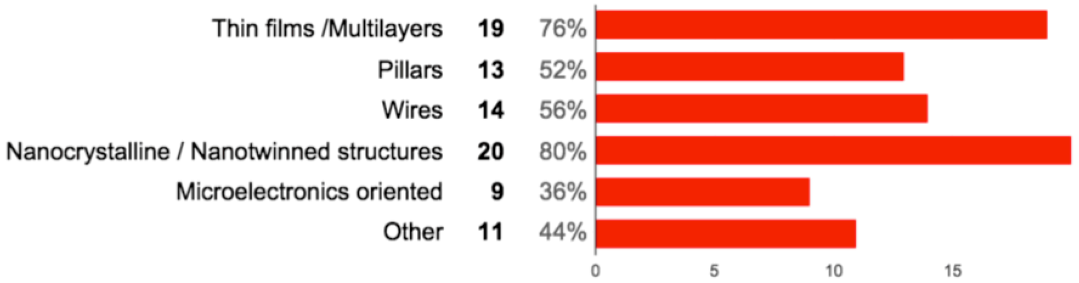

Figure 1. Objects of interest. Several answers possible. The chart shows the number of positive answers, followed by the corresponding percentages.

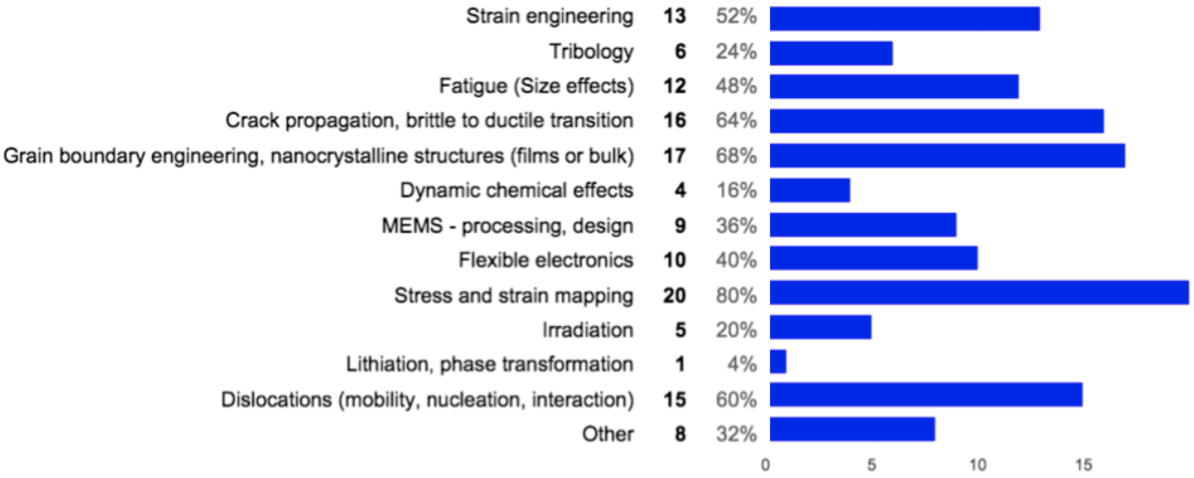

Figure 2 Topics of interest.

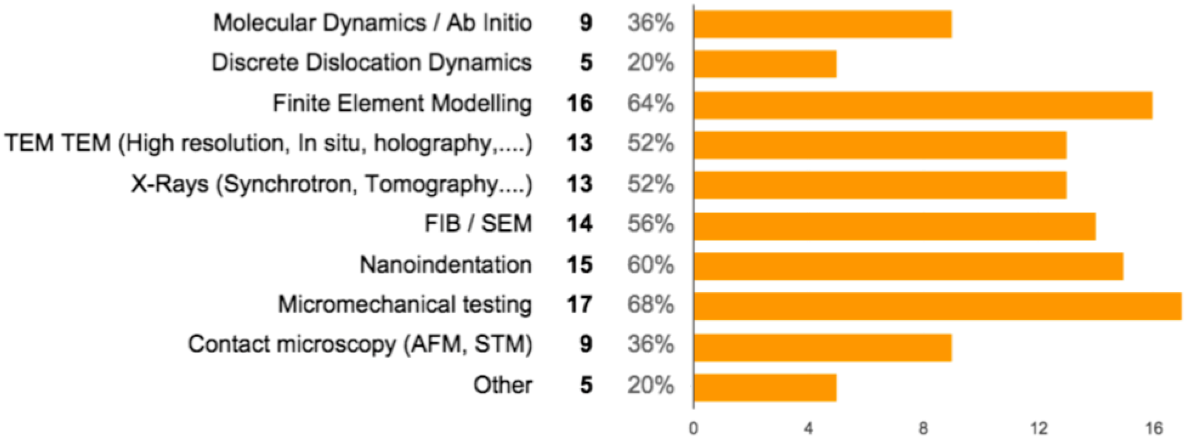

Figure 3 Methods of interest.

The poll has been filled by 26 laboratories throughout France and Europe plus 2 additional ones coming from the US (see below the list of potential participants §3).

As expected, the **objects of interest** (Figure 1) of the network combine historical important ones (thin films, multilayers), those that have emerged recently (micro and nanopillars). Nanowires constitute a re-emerging trend as the capabilities to investigate them (X-Rays, micromechanical testing) allow accessing their mechanical properties with an unprecedented accuracy. The demand to focus on bulk nanostructured materials (nanotwinned/nanograined) will have to be taken in account.

The **topics of interest** (Figure 2) also reflect the combination of the core interest of the network (Strain engineering, stress and strain mapping, dislocations), as well as considerations in relation with emerging objects of interest (bulk nanocrystals, nanotwinned metals for instance). Associated with this, grain boundary engineering, crack propagation and fatigue, some classical themes of physical metallurgy. A couple new themes related to the mechanical properties of the nano-objects have been proposed (lithiation, dynamical chemical effect-diffusion), but they did not receive many votes and will probably remains peripheral to the network.

One of the strengths of the Mecano network is its real ability to have experimentalists and theoreticians working together. Again, this shows in the above Figure 3, in which **methodologies** relies both on simulations and experiments. Finite element calculations can be seen as an unavoidable technique that is used both by mechanical theoreticians and experimentalists, especially when techniques such as nanoindentation or micromechanical testing (pillar, micro beams,) often require to know how stress and strain are spread in the structures under test. But the key advantage of working on small structures is that both the simulation box (in Molecular Dynamics for instance) and the actual device under test can be of similar dimensions, which represents a serious step to convergence between simulations ad experiments.

These aspects will be developed in the scientific objective section, below, §2.

2. Scientific objectives of the network

Size effects in condensed matter physics have both industrial and fundamental issues. The reduction of transistor size and their implemented integration on Si wafers has already raised issues of increased metal resistance in small dimensions for instance. Fundamentally, the impact of geometrical confinement on the mechanical properties has been known for decades. Thin films are for instance stronger than their bulk counterpart, and whiskers showed size effects already in the 50' ([1,2]……). But the micropillar test that appeared in 2004 ([3,4]) revealed that this size effect, spectacular at the nanoscale, could extend far in the micron scale. What appeared as a major revolution 10 years ago has expanded to an almost routine test on all kinds of systems (ceramic, biocomposites, metals, semi-conductors) but is still highly debated today. Intriguing effects such as avalanche type behavior [5], [6] are for example poorly understood. But a beneficial side effect of this new test was to renew the interest in small-scale objects and to explore an unknown region of physical metallurgy.

Because of the broad area swept by the members of the network, and because **topics**, **objects** and **methods** give many reading entries to this area, we propose to keep 4 axis, as in the previous network, but to modify them slightly in order to cope with the expressed will of the participants. The goal is of course to have these four axes mixed and interacting with each other as much as possible.

The proposed 4 axis of the next GDRi MECANO are:

- **Growth and processing of micro and nano-objects**: effect of stress, composition, processing route

- **Experimental methods**
  - Stress and strain mapping: TEM, X-Rays, contact,…
  - Mechanical testing: AFM, nanoindentation, micropillar, nanobeams, microtensile…

- **Modeling and simulation**:
  - Mesoscale methods: Finite Element, Discrete Dislocation Dynamics,
  - Atomic scale methods : Molecular Dynamics, Ab initio

- **Mechanisms at small scale**: stress relaxation, dislocation, Grain Boundary (GB) engineering, twinning, crack propagation

The aim of the following state of the art is to present some striking, possibly new, publications in the field of the mechanics of nano-objects. This presentation is involving a significant portion of publications from the network members, but is neither exhaustive nor objective. Its goal is also to reflect the true global and dynamical effort the world of research is putting together to solve the present and intriguing questions of size effects in mechanical behavior of condensed matter at small scale.

2.1. Growth and processing of micro and nano-objects

Several methods have now gained atomic or near atomic control to create nanostructures where metastable states are maintained by the reduced volume. This is obviously the case for epitaxial systems, but core-shell nanoparticles created by physical or chemical ways demonstrate physical and structural properties that cannot be attained in bulk structures [7,8]. Recent advances in imaging using AFM [9], optical [10] and TEM [11] allows to observe the mechanisms at play during growth, giving unprecedented insight about the stress and strain state of the created objects. Metastable structures can also been undone, creating islands by dewetting, which is another way to create small scale objects [12], [13].

The way nano-objects are processed is obviously key to their subsequent mechanical properties as those are heavily influenced by unusually high surface/volume ratio. Chemical composition, native oxides or implanted layers, resulting from the processing route have been found to have dramatic effects. Ga implantation during FIB-ing of micropillars can for instance divide the strength of a metal by almost 10 [14].

In small-grained materials, where strength and ductility usually follow opposite ways as the grain size is reduced, methods such as severe plastic deformation, powder compaction, vapor deposition, may result in materials with similar sized microstructure and different properties [15], [16].

Microelectronics paved the way for the ultimate control of size, chemical composition and strained objects on Silicon [17]. People are now projecting the use of strained nanowires to increase the semiconductors efficiency [18-20].

Lithography also expanded to the fabrication of so-called MEMS (Micro-electromechanical Systems), that can be both testing units, samples to be tested or a mix of both ([21-23], [24,25]). There too, the processing routes have often been overlooked and materials science input may be beneficial to correlate structure and properties.

2.2. Experimental methods

2.2.1. Imaging stress and strain

Stress fields are often converted from strain measurements using the laws of elasticity, and most of the studies show that this is a good approximation down to the nanometer size or when strains are on the order of a percent. Beyond these limits, inter-atomic interactions and elasticity becomes non linear, and these frontiers are of great interest for the Mecano community.

In the domain of X-rays, the most striking progress concern coherent x-rays [26], [27], as this technique allows determining complete stress states of nano-objects [28] or small objects containing a small amount of defects [29] [30]. This is obviously a fast and promising new field, boosted by the performance of coherent and sub-micron beams available at synchrotrons, where many members of the community are involved.

Electron microscopy (transmission – TEM or scanning SEM) also improved in the last years due to better sources, aberration correction systems, low voltage imaging, diffraction mapping, chemical detection and 3D reconstruction softwares, [31] [29] [32] Electron holography also opened a path to very precise strain mapping, adding to the already high resolution capabilities of TEM [33] a field of view that is now allowing mapping on micron-scale areas[34]. One of the current challenges in this field is to combine this technique with variable fields inside the TEM, including stress fields [35,36]. A key factor for implementing in situ holography relies on the speed and definition of cameras, and speed larger than 100 images/seconds become available with direct electron detectors or fast electronic for classic CCD chips.

2.2.2. Mechanical testing

This is a field where spectacular progresses have been made in the past years. Especially in combining techniques that were distinct before. Sensors have gained in compactness and precision, which allows the integration of measurements in or under observation tools. "In situ" has thus become a standard word as most of the mechanical testing experiments can now be performed under a micro-beam at the European Synchrotron, in a TEM or using a confocal microscope. At the same time, the time and spatial resolution of the microscopes, detectors reached unprecedented accuracy.

Because of its apparent simplicity, indentation tests are extensively used to probe a material or a surface strength, even if the translation of hardness into more simple parameters such as elastic modulus or yield stress may require modeling. One of the most cited paper in materials science by Oliver and Pharr [37] (more than 14k citations) describes how to extract such parameters from a test. Since then, instrumented indentation (or "**nanoindentation**"), a technique able to retrieve a force vs displacement plot has completely transformed the field in a way to probe any material [38,39] at a very local scale, and, by extension to perform mapping [40]. And several technical implementations have moved from the laboratories to industrial companies[1], still very involved in research, but able to offer stand-alone systems. Size effects were detected early in nanoindentation as the probed volume diminishes with the indentation depth [41]. On a theoretical point of view, nanoindentation has brought insight on the nucleation of incipient dislocations [42], [43], [44] and offers a very convenient way to localize plasticity for further analysis. Recently, this technique upgraded to high temperature capabilities, opening yet another area to explore, as attested by the exponential growth of publication in the field [45].

In parallel, nanoindenters, with their capacities of measuring force and displacement were turned into versatile **micromechanical testing** units for a number of small scale objects such as wires,

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[1] Hysitron, Keysight (previously Agilent) , Anton Paar (previously CSM instruments), Nanomechanics inc. are some of the leading companies for nanoindentation instruments. They often sponsor and support meetings linked to nanomechanics, and we hope they will extend this to the GDRi Mecano…

pillars, beams, chevrons, dog bones samples.... The various configurations allow one to suppress strain gradients [46-48] or to use them in a controlled way (localizing plastic deformation, inversing stress direction, [49-51] [52,53] [54]. FIB-SEM (Focused Ion Beam – Scanning Electron Microscope) machines are ideal tools to develop suchs test. Not only the machining possibilities at the micron and nanoscale seem endless, but the high accuracy of SEM imaging allows a precise strain measurement of the sample under test [55,56] [57].

MEMS-based platforms can also help miniaturizing even more such testing units [58] and are now serving as support for very small wires [59], [53], [60], especially when temperature is needed or when only compression is available [36,61]. Very small forces can also be attained using electrostatic devices [62,63], bridging the gap with contact measurements attainable using **AFM** (Atomic Force Microscopy) [64] [65]. Other techniques specific to thin films (bulge testing [66] for example) could be cited here, but for the sake of space, only the most popular one were mentioned.

Scientists and engineers face multiple challenges here: increasing the acquisition frequency to a point where intermittent plasticity does not make the system diverge, imposing actual strain rates, imposing and measuring temperature, and, at the same time leaving enough space for observation in the case of in-situ platforms. The goal is of course to observe intrinsic effects and avoid artifacts due to dimensions shrinkage.

2.3. Modeling and simulation

It was once very difficult to link the model of a moving dislocation calculated in a nanometer-sized simulation box to the behavior of a cm-sized sample because the time and dimensional scales to bridge were simply too far away. If a large time gap remains between real life experiments and computer-based calculations, the size reduction of the objects under test constitutes a real advantage in trying to match theory and experiments. The perfect example of such a match concerns the theoretical strength of crystals. First based on a simple model (also called Frenkel's model) of sinusoidal potential, atomic interactions in a crystal predicted strength on the order of $G/2\pi$, G being the shear modulus of the lattice ([67]. Until very recently [68], this strength has never been attained because of the always high dislocation content of metals or because surface flaws and/or imperfections in the testing unit. At the same time, people performing ab initio calculations on perfect crystals [69] or looking for the first **dislocation nucleation** event [70] systematically computed stress levels comparable with this theoretical stress. G/10 is now observed in FCC metallic whiskers, and samples and experimental set-ups have reached a precision such that activation energies, anelasticity and probabilities are in line with those revealed by simulations [71], [72], [73], [74]. Nucleation has also been heavily simulated in the case of the nanoindentation of a free surface [75] [76] [77].

Once the dislocations are created, their **motion** can be subject to friction forces that are thermally activated and that complicate the deformation of crystals with BCC structures such as Fe [78] [79] or HCP such as Ti, Zr [80]. Recent developments show that quantum effects may expend much further than at absolute zero temperature. The competition between these intrinsic obstacles to dislocation motion and their extrinsic confinement may lower or erase the size effect [81,82].

2.3.1. Atomic scale methods

Simulations using extensive **Molecular Dynamics** (MD) have also tackled more complex confinement problems such as the plastic deformation of nanocrystals where competing mechanisms (dislocation nucleation, absorption, GB motion GB diffusion…) can coexist. [83-87]. However the use of a given set of potential and "experimental" conditions where the system is forced through a deformation path at very high strain rate may exclude some of the potential mechanisms and return a complex picture even for the early stages of deformation.

Finer methods such as **Nudge Elastic Band (NEB)** where energy paths are carefully calculated proved much more precise in determining acting mechanisms. [88] [89] [90]. They have been employed for about 10 years but require more computation time. Recently, such a method has been successfully validated for the motion of disconnection in the so-called shear-coupled grain boundary migration, an alternative process to dislocation plasticity [91]

As for force sensors, there is an almost continuum between atomistic simulations (Ab initio, molecular dynamics) and macroscopic Finite Element methods. All these methods require hypothesis and approximations. Comparing methods is not always done [92], but such discussions have already started in the Mecano network and proved beneficial for the whole community (theoreticians and experimentalists). Hypothesis (quasi static states, strain rates, box sizes, energy paths…) are for example better defined, making comparison with experiments more fruitful. The reason why two opposite solutions can be returned for a similar initial situation (e.g. dislocation twin interaction [93,94]) show that the devil is in the details. And sometimes also in the use of interatomic potentials for molecular dynamics.

Ab initio calculations overcome this problem of fitted potentials, but they are still too greedy in calculation time to grasp dislocation-dislocation or dislocation-GB interactions where long range fields are needed. They can however fuel mesoscopic models in accounting for dislocation segments mobility or for the stability of a given configuration [95].

2.3.2. Mesoscopic methods

Interaction mechanisms may stand at the core of the plasticity mechanisms in small structures and quantitative values are sometimes missing, especially for **Discrete Dislocation Dynamics models (DDD)** where a given dislocation structure interacts with itself or surrounding obstacles. Such modeling has proven extremely precise for capturing very complex behaviors such as fatigue [96], and at small scale, microbeams behavior [97] or intermittent sources and stress oscillations [98]. The combination of DDD and **Finite Element Method (FEM)** also proved very efficient to obtain meaningful stress and strain fields in the case of long range interactions [99].

2.4. Mechanisms at small scale

Linking the micro or nanostructure of structure to the observed properties is central to materials science and it makes no exception at small scale. Size effects have often been highly debated, and the Hall-Petch effect [100,101] makes no exception. This empirical law established in the 50's states that the yield stress of a metallic polycrystals increases as the inverse of the square root of the mean grain size. Physically, this has been explained by the piling up of dislocations or the volume needed to nucleate fresh dislocations [102-104]. However at the nanometer scale, this law breaks down and other mechanisms are supposed to take over.

As mentioned earlier (section 2.3.1), atomic scale simulations have started tackling this problem many years ago. One of the features put forward as a relevant alternative mechanism to dislocation plasticity is called shear-coupling GB motion. Such a mechanism has been known for decades, but because GB descriptions are not unique, and because very few people are studying it experimentally, the mechanism is very poorly known and a single theory rules the community [105]. Recently, in situ TEM experiments [106,107] revealed that disconnections [108] are governing this type of GB-based mechanism. New simulations blame the same suspect [91], but also open for a complete rehabilitation of the main theory of GB migration.

Dislocation nucleation and multiplication are also two key mechanisms in defect-depleted structures such as single crystalline wires and pillars. As stated before, nucleation processes seam in line with elasticity and plasticity laws and multiplication processes are also found to occur in a very classical way down to 100 nm[109], [110]. Such a low limit was not expected, but proves the solidity of the dislocation theory. Using this theory, it is now conceivable to quantify size effects in structures with very low defect densities [111]. In fact a unified approach is now able to grasp size effects due to microstructure (poly and single crystals, metals or ceramics….) in a very elegant manner [112,113]

Twinning and detwinning processes have been among the highly debated topics in the last 5 years or so [114-116], and there too, some of the believed rules seem shaken up by surprising results [117]. Especially, researchers are tackling the problem of partial dislocation glide and twinning in non FCC structures.

The "dark side" of plasticity is called fracture and there too, size effects play a very intriguing role, either through grain size [118] or interfaces [119]. Brittle to ductile transition has been investigated in compression for semiconductors [120,121], silica [122] or bones [123]. The absence of microstructure (silica) or the absence of dislocations (thus requiring nucleation at very high stresses) are extreme cases of toughness testing. Because compression testing is not prone to test a material toughness, advanced tests using notched specimen milled by FIB may be a way to investigate this paremeter with techniques compatible with micro-pillar testing [54].

3. Partners and coordination

The following list of partners has been established based on the poll initiated inside the previous Mecano Network, and from previous partners. It will be completely open to new participants along the 4 years of the GDRi. Compared to the 2012-2015 GDRi, two American laboratories have joined this proposal. The 28 French, European and US laboratories that have filled this poll and thus expressed their will to renew their participation to Mecano are indicated in italics. Those from the preceding network are in plain text. In any case, the network is not close and definitely open to good will and late applicants.

3.1. European and American partners

- *Université Catholique de Louvain, Belgique (Thomas Pardoen)*
- *Erich Schmid Institut , Montanuniversität, Leoben, Austria (Daniel Kiener)*
- *EMPA, Thun , Switzerland (Johannes Michler)*
- *Max Planck Institut, Dusseldorf, Germany (Gerhard Dehm)*
- *Georgia Institute of Technology, Atlanta, GA, USA (Olivier Pierron)*
- *University of Pensylvania , Philadelphia, PA, USA (Daniel Gianola)*
- *Max Planck Institute, Stuttgart, Germany (Günther Richter)*
- *Max Planck Institute for Intelligent Systems, Stuttgart, Germany (Günther Richter)*
- Oxford University, UK (Angus Wilkinson)
- *Paul Scherrer Institute, Switzerland (Helena Van Swygenhoven)*
- Paul-Drude-Institut für Festkörperelektronik, Berlin Germany (H. Riechert)
- *Erlangen University, Erlangen, Germany (E. Bitzek, M. Göken)*
- *Chair of Materials Science and Methods, Saarland University (C. Motz)*
- *Physical Metallurgy, TU Darmstadt (K. Durst)*

3.2. French partners

- *Centre des Matériaux, CNRS Mines Paris Tech (H. Proudhon)*
- *CEMES CNRS, Toulouse (A. Ponchet, M. Legros)*
- *CINAM CNRS, Marseille (P. Müller)*
- DEN\DEC\SESC\LLCC CEA-Cadarache (C. Sabathier, H. Palancher)
- *FEMTO-ST CNRS-Univ. Besançon, Besançon (F. Amiot)*

- IEMN CNRS-Univ. Lille 1, Villeneuve d'Asq (A. Devos)
- Institut Jean Lamour CNRS Ecole des Mines de Nancy (A. Mezin)
- INAC CEA-Grenoble (O. Robach)
- *ILM CNRS-Univ. Claude Bernard, Lyon*
- INL CNRS-Centrale Lyon, Ecully (G. Grenet)
- INSP CNRS-Univ. P. et Marie Curie, Paris (B. Croset)
- *IPR CNRS-Univ. Rennes*
- IS2M CNRS-Univ. Haute Alsace, Mulhouse (D. Dentel)
- ISIR CNRS-Univ. P. et Marie Curie, Paris (S. Regnier)
- *IM2NP CNRS-Aix-Marseille Universités, Marseille (O. Thomas)*
- IMEP-Lahc CNRS-Grenoble INP-UJF-Université de Savoie (L. Montes)
- Labo Claude Goux –CNRS Ecole des Mines de Saint-Etienne (C. Maurice)
- *LEM CNRS-ONERA, Chatillon (B. Devincre)*
- LETI CEA, Grenoble (P. Gergaud)
- LMA CNRS-Univ. de Provence (F. Lebon)
- LPMCN CNRS-Univ. Lyon 1, Villeurbanne (A. San Miguel)
- *LPMT CNRS -Univ. Haute Alsace, Mulhouse (M-H. Thuillier)*
- *LPN CNRS, Marcoussis (F. Glas)*
- *LSPM CNRS-Univ. Paris XIII, Villetaneuse (D. Faurie)*
- LTDS CNRS-Ecole Centrale, Lyon (T. Hoc)
- *MATEIS, INSA-CNRS, Villeurbanne (K. Massenelli -Varlot)*
- MPQ CNRS-Univ. Paris VII, Paris (I. Favero)
- *PPRIME CNRS – Poitiers (L. Pizzagalli)*
- *SIMaP CNRS-Grenoble INP, St Martin d'Heres (M. Verdier)*
- *SIMM- ECPCI-CNRS-Paris (E. Barthel)*
- Saint-Gobain Recherche, Aubervilliers (A. Benedetto)
- STMicroelectronics (H. Jaouen)

3.3. Coordination - Scientific Committee

As of today, the Scientific Committee is not decided yet and its creation will depend on the acceptance of this proposal by the CNRS and the signature of the various partners. Legally, "*a Scientific Committee will be made up of representatives of the Network Parties. The Scientific Committee is composed of sitting representatives from member laboratories/institutes/centers, appointed by the Party (or Parties) to which the laboratory/institute/center is affiliated.*" In practice, a reduced committee will be created, in agreement with all the parties. One or two co-coordinators emanating from each axis (as defined in §2) should also assist the coordinator.

The Scientific Committee will meet at least once and possibly twice a year to decide the place, topics and program (invited speakers) of the forthcoming meetings, workshops and schools.

4. Network activity : meetings, workshops and schools.

The network is aimed at promoting scientific discussions, exchanges between the members in the field of mechanics of nano-objects. For that purpose, the next GDRi will organize various events:

- General meetings open to all the topics of the network;
- Dedicated workshops on specific topics considered as timely;
- Schools bringing together scientists and graduate students where basics are reviewed and recent research results are put in perspective.

On average the previous network has been organizing every year one general meeting and two events, either workshop or school. The next meeting will try to keep the same basis.

4.1. General Meetings

One general meeting will be organized per year, in European locations. We are also targeting a symposium devoted to the mechanics of nano-objects at the Fall Materials Research Society general meeting (possibly in 2017 or 2018) or at the TMS annual meeting in the US. Several members of Mecano have already organized such symposia and this will allow a full involvement of our US partners, and the possibility to better advertise the network activities abroad.

4.2. Schools.

At least one graduate student school will be organized during the 4 years period. Basic notions (elasticity, mechanical testing, dislocations, X-Ray diffraction…) will be taught as well as state of the art knowledge in the field of nanomechanics

4.3. Workshop

Several workshops with more focused topics will be organized. A rhythm of 1 or 2 workshop per year is expected. They will have to be initiated by network members. These workshops can target a specific technique, topic or type of sample.

4.4. Website, administration.

The current web site (**http://www.im2np.fr/GDRI_CNRS_Mecano/index.html**) is hosted at IM2NP and managed by Cathy Paitel. The next one will be hosted by CEMES in Toulouse and managed by M. Legros and Jean-Noël Fillon (CEMES). Rose-Marie Melendo (CEMES) has also accepted to take in charge the administrative part of the network (Accounting).

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