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# Mechanical characterization of materials at small length scales †

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## Abstract

A review on the mechanical characterization of materials at small length scale is presented. The focus is on the different micro- and nanoscale testing techniques, the variety of materials investigated by the scientific and industrial communities and the mechanical quantities identified by such methodologies.

*Keywords*: Mechanical characterization; Mechanical properties; MEMS; NEMS

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## 1. Introduction

The interest on materials behavior at small length scales has gained considerable attention in the last two decades, as a consequence of the increasing application, production, and commercialization of various kinds of micro- and nano- electro-mechanical systems (MEMS/NEMS). Due to their small size, short time response, high performance and low energy requirements, these devices are currently used in a variety of industrial, consumer and biomedical applications [1-4]. Such success has stimulated a further improvement of their design, in order to produce even more reliable and competitive components.

The materials used by the MEMS and NEMS industries are not only the typical silicon-based materials of microelectronics, but also metals, alloys and polymers (e.g., polyamide). Furthermore, different kinds of nanostructured materials are being increasingly used.

A knowledge of the mechanical properties of such materials is essential for designing, fabricating and predicting the reliability of microdevices. In addition, with the further spread of MEMS and NEMS, tighter design constraints enforced by the economic competition will necessitate the development of new materials and innovative fabrication processes. As a consequence, reliable and repeatable evaluation of the mechanical properties of both currently used and emerging materials is needed. In this context, standardized methodologies for characterization should be defined.

Unfortunately, the standard well-assessed techniques for mechanical characterization at the macro-scale cannot be transferred to the micro- or nano-scale, since the machinery and equipment they involve are not suitable for manipulating components with submillimeter size. In addition, it has been shown that the mechanical properties of materials at smaller length scales cannot generally be derived from bulk properties determined by ordinary macro-scale methods [5, 6]; the fact that material properties change with specimen size has been well known for several years [7, 8]. As a consequence, testing materials at small length scales involves very small specimens and miniaturized testing structures. Such testing structures should be equipped with high resolution measurement systems for accurately measuring loads and displacements. To match such requirements, new and appropriate techniques and equipment have been developed.

Very few studies on mechanical characterization of micro- and nano-components were published before the 1980s, but since the advent of MEMS, the number has dramatically increased.

The histogram reported in Fig. 1 shows the trend of scientific articles on the mechanical characterization of materials at micro and nanoscales published since the 1920s. Filled bars are related to the global production of manuscripts including work dealing with both the micro and nano length scales[1], while empty bars represent the production related to the nanoscale only. Studies regarding nanometer length scales are increasing, but not as rapidly as those regarding micrometer

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[1] Here, by microscale tests the authors refer to those carried out on samples with characteristic length on the order of micrometers. Similarly, by nanoscale tests the authors refer to those carried out on samples with characteristic length on the order of nanometers.

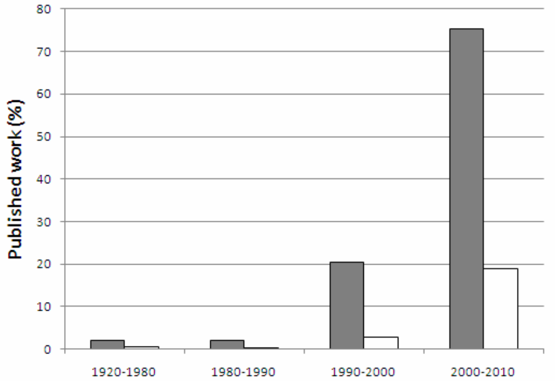

Fig. 1. The trend of the production of scientific articles about the mechanical characterization of materials at micro and nanoscales since the 1920s: in gray the global production; in white the production related to nanometric length scale.

length scales, which reflects the fact that while MEMS are commercially available, NEMS are not yet.

Several testing methods for material samples with characteristic size ranging from few nanometers to several hundred micrometers, have been proposed, developed, and employed for determining mechanical properties such as Young's modulus, Poisson's ratio, yield strength, fracture strength, hardness, and endurance limit.

In this review we summarize, classify and compare all of these techniques. We establish what are the most used, why they are so popular, and what is the actual vogue. Furthermore, we identify the most investigated materials and how the interest in them has changed with time.

## 2. Mechanical testing at the micro- and nano-scales

Material mechanical properties are fundamental input parameters for structural design of micro- or nano-devices. The accuracy of numerical modeling and simulation results depends on the accuracy of the material properties provided as input. For this reason, during the last decades many suitable techniques have been developed to improve the measurement accuracy and/or to increase the number of the determinable mechanical quantities. We present a classification of these techniques in Fig. 2.

A requirement for carrying out accurate mechanical characterization at the micro/nanoscale is the requirement of high-resolution systems for displacement and force measurements. For testing micro and nanostructures, many microscopy techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM), have been widely utilized. In fact, these instruments provide useful ways of measuring dimensions and deformation with nanometer resolution. Regarding force measurements, custom-made transducers are often designed, even if commercial load cells are also available.

Many of the testing systems proposed in the literature have been assembled by the researchers who designed them. Some of those have also been patented in the last ten years [15]. However, some companies, like DTS (Menlo Park, CA) and Zwick (Ulm, Germany) have also released to the market complete stages to perform tensile tests on micro/nanosamples [16, 17].

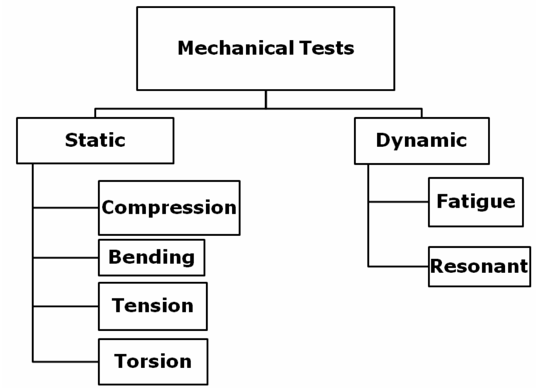

Fig. 2. Classification of the test methods for mechanical characterization of materials at micro- and nanoscales.

In the following sections, quasi-static tests and dynamic tests are discussed in more detail.

## 3. Quasi-static tests

### 3.1 Tension tests

The goal of micro/nanoscale tension tests is to obtain elastic and plastic mechanical data by fixing a specimen at one end and applying increasing tensile load at the other end.

Uniaxial tension tests have the advantage of applying uniform stress and strain fields to the specimen. Such a feature makes it easy to employ and is the reason why it is so widely used to determine mechanical properties at larger scale. However, performing tension tests at micro/nanoscale is challenging because of the difficulties in preparing free-standing and stress-free specimens. Furthermore, the requirements for sample alignment, deflection and load measurements are stringent, and in the case of brittle materials, fracture induced by specimen gripping may be an additional problem.

Various arrangements have been used in tensile testing of micro/nano-specimens differing in the fabrication and gripping of specimens, systems for force and displacement measurements, and aligning procedures. For example, specimens in frame [18], fixed at one end [19], separated [20] and co-fabricated with the loading and measuring systems [21] have been used. Gripping of specimens has involved frictional [22] and electrostatic attraction forces [23], glues [24], tapes [25] and connecting ring [26].

With respect to force measurements, commercial loading cells can be chosen, but, usually, commercial transducers cannot achieve nano-Newton resolution. Therefore, researchers very often employ home-made force transducers such as cantilevers [27], microfabricated frames [28], flexural spring [21] and strain gauges [29].

Accurate measurements of the extension or the strain can be carried out using: commercial capacitance-based displacement transducers, measurements of shifting in interference pattern [18], diffracted spots [30], digital image correlation method [31, 32], optical encoders [29], Moiré method [33, 34], electron pattern speckle interferometry [35], or microvernier gages [36]. Tensile tests are usually performed under displacement control, which can be provided by different actuators, like piezoelectric [37], inchworm [38, 39] and thermal actuators [40].

In tensile tests an important aspect is the correct specimen alignment along the loading direction. A slight misalignment may result in an undesired bending action, which could cause a non-uniform stress distribution, affecting the effectiveness of the test [41]. The alignment requirement can be accomplished by visual inspection using optical microscopes and precision positioning stages [42] or high-resolution microscopes in conjunction with nanomanipulation tools [43].

In order to circumvent the aforementioned problems, several researchers [44, 45] developed "on-chip" test devices, which are MEMS-based actuating/sensing systems. In the first generation, the samples were co-fabricated during the microfabrication process together with all the mechanical parts needed for loading the specimen and for measuring displacements and strains. However, those testing devices could not be used to perform more than one test. Furthermore, they were not suitable to test all kind of materials, but only those that could be deposited according to microfabrication limits. Therefore, they could not be used to test 1D nanostructures, like nanowires and nanotubes. For this reason, the most recent on-chip testing devices are designed to test samples, that can be separately fabricated and then placed on the loading frame. However, for such tests it is challenging to provide correct alignment of the specimen.

On-chip testing devices include on the same MEMS platform actuating mechanisms (e.g., thermal actuator [46, 47] and electrostatic comb drives [48]) integrated with sensing capabilities (e.g., capacitive sensing). Such devices are probably the most powerful for studying deformation mechanisms by real-time imaging of defect nucleation and propagation, and they have been used for *in situ* mechanical testing of nanostructures within a SEM or a TEM [28, 49] leading to very successful results, sometimes on truly nanoscale specimens. Another advantage of on-chip testing methods is the availability of standard microfabrication techniques to fabricate them. Furthermore, since they are electro-mechanical systems, they could also allow performance of coupled electro-mechanical tests [50]. On the other hand, they require sophisticated design and calibration analyses. In addition, sometimes the forces developed by on-chip actuators are insufficient to break the specimens in quasi-static condition. It is worth mentioning an interesting attempt made by some researchers to simplify their testing device by proposing a test fixture without the loading actuator [51]. In fact, the load on the specimen is applied by exploiting the internal stress affecting one material beam.

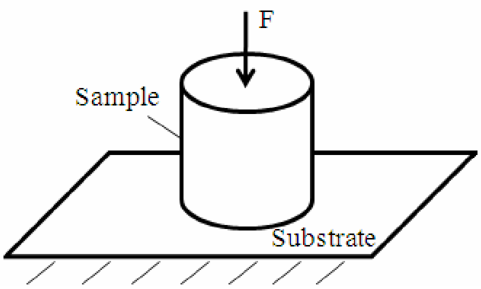

Fig. 3. Compression test.

Another limitation of the on-chip testing systems regards the load sensors. In spite of what happens at macroscale, load sensors used in MEMS-based tests have very low stiffness, in order to guarantee high load resolution [50]. However, this can cause instability phenomena when the stress in the sample has drops, which could result from phase transformation [52].

Finally, tensile tests were adopted for determining the size of the representative volume element (RVE) of materials, e.g., polycrystalline silicon [53], or particulate composites [54]. From the design point of view, the knowledge of the REV size can play a major role, since it defines the scale limit for which MEMS/NEMS devices can be properly designed by using the bulk material properties.

### *3.2 Compression tests*

The compression test is mainly used to study plasticity and to investigate mechanisms that are responsible for the higher strength of materials at micro/nanoscales [55].

The first compression test at the microscale [56] was performed on micro-pillars (Fig. 3). From a technical point of view, such a test is simpler to implement than the tensile test, because sample gripping is less complex.

Micron or sub-micron pillars (with small aspect ratio to avoid buckling) can be fabricated by focused ion beam (FIB) and loaded by a flat head nanoindenter [57, 58]. This test can be performed up to very large strains in order to investigate not only the first instants of plasticity but also the large strain behaviour and strain hardening response. Recently, nanocompression experiments have also been conducted *in situ* within a transmission electron microscope (TEM) for real time imaging of tests [59].

Based on numerical simulations, guidelines for the implementation of micro-compression experiments on various materials including isotropic materials (such as nano-structured materials and metallic glasses) have been suggested in Ref. [60].

### *3.3 Bending tests*

Alternatively to tension tests, mechanical properties of materials at micro/nanoscales can be obtained from bending tests,

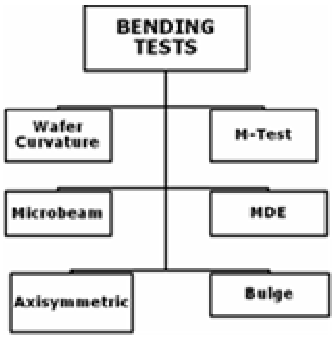

Fig. 4. Bending tests for mechanical characterization of materials at micro- and nanoscale.

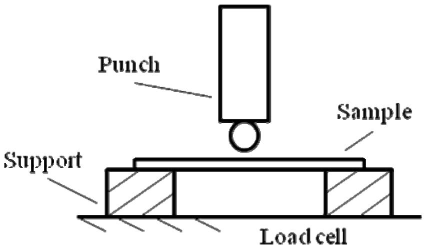

Fig. 5. Axisymmertric test.

which are sometimes preferred over tension tests, since they require smaller forces and produce larger displacements. On the other hand, they allow one to determine a smaller number of parameters, including the elastic constants (Young's modulus and Poisson's ratio), the residual stresses, and the yield and fracture strengths. Such quantities are typically determined by inverse procedures exploiting analytical models.

Often, the boundary conditions required to use these models do not apply in real experiments because of fabrication limits and, as a consequence, errors can be present in the solutions.

Different test configurations involving different loading fixtures, measurement equipment and specimen geometries, have been proposed (Fig. 4). The most prominent are: axisymmetric tests, microbeam tests, bulge tests, MDE tests, M-tests, and wafer curvature tests, and recent on-chip bending tests, all which will be described below.

### 3.3.1 Axisymmetric bend test

In this test, the circular specimen is placed on a hollow cylindrical support and loaded at its center by a spherical indenter (Fig. 5).

The resulting axisymmetric biaxial stress field ruptures the specimen on the opposite side of the load application point. This allows for the evaluation of the fracture strength by using a suitable formula relating the fracture stress at the center of specimen to the applied load. Although this test has the advantage of easy to fabricate specimens, some aspects limit its use: the difficulties related to the precise positioning of the specimen; the uncertainties on the boundary conditions; and it provides information only about the fracture resistance.

In the past it was employed for analyzing freestanding thick silicon plates [61, 62].

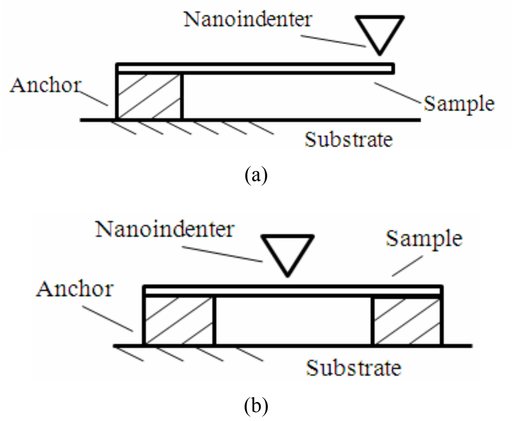

Fig. 6. Microbeam tests.

### 3.3.2 Microbeam test

The specimen is a rectangular beam loaded with a concentrated bending force. It can be cantilevered or clamped at both ends. In the first configuration, the load is applied at the free end of the cantilever (Fig. 6(a)), while in the second configuration, the load is applied at the middle of the beam span (Fig. 6(b)).

Such tests can be used for determining the Young's modulus or the fracture resistance of the material sample. In particular, the Young's modulus can be derived from analytical models involving geometrical quantities, elastic properties, applied loads, and displacements, after recording the beam deflection as a function of the applied load. Loads can be applied by different means: an AFM [63], a probe attached to a micro-force testing machine [64, 65], or electrostatic actuation [66, 67].

One of the limits affecting this type of tests is related to the inefficacy of the boundary conditions, since it is very difficult to reproduce the ideal boundary conditions with rigid constraints in real testing configurations. Furthermore, the right choice of the beam length also can be crucial (e.g., if the beam is too long, the force required to bend it is small, and so the device is difficult to calibrate, whereas if the beam is too short, the required force is higher, but more complex analytical models have to be used [68]). However, this test is very common since freestanding beams can be fabricated by ordinary microfabrication methods.

### 3.3.3 Bulge test

This technique was introduced by Vlassax & Nix in 1992 [69]. The specimen is a free-standing membrane (circular, square, or rectangular in shape), which is clamped around its periphery to a supporting frame (Fig. 7).

Pressure is applied to one side of the membrane, for instance by compressed air [70]. The resulting deflection at the center is recorded as a function of the applied pressure (e.g., with a laser interferometer [70]).

With such a technique, it is possible to derive mechanical properties of the material sample as the biaxial stress-strain

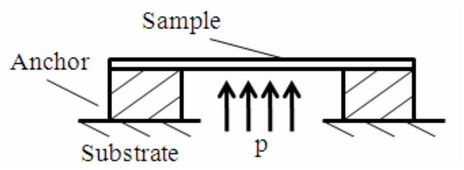

Fig. 7. Bulge test.

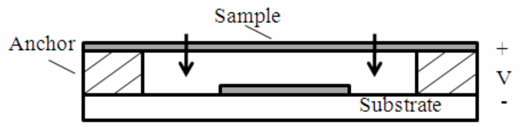

Fig. 8. M-test.

curve and the yield strength, and to investigate plasticity size effects [71].

The Young's modulus, Poisson ratio, and residual stress field can be extracted using continuum mechanics models.

The accuracy of the results can be affected by the difficulties in reproducing the ideal boundary constraints, which are compliant instead of infinitely stiff, and, more generally, to the uncertainties related to the knowledge of the geometrical entities. Furthermore, it can happen that during the test the membrane separates from the substrate [68].

Nevertheless, because it is relatively easy to fabricate membranes using ordinary micro fabrication tools, this technique is commonly used.

### *3.3.4 M-test*

The specimen is a microbeam, loaded by an electrostatic force (Fig. 8) until the onset of instability phenomena (i.e., collapse).

The voltage causing such an instability (called the pull-in voltage) is directly related to the elastic properties and the residual stresses of the material sample. In particular, for ideal geometries (i.e., neglecting residual strain and compliance of the supports), some closed form expressions have been reported [72]. However, these expressions can be appropriately modified to take into account non-idealities introduced by the fabrication process [73].

By this test both the Young's modulus and the residual stress field can be derived.

### *3.3.5 Wafer curvature test*

The wafer curvature test is used to evaluate the residual stress field inside constrained thin and thick structures, in particular, continuous films deposited on thick substrates [74]. This method relies on Stoney's formula, which relates the residual stress inside the film to the substrate-film curvature, under the assumptions that the film is much thinner than the underlying substrate and that small deflections occur [75]. So, residual stresses can be computed by measuring the film thickness, the substrate elastic properties (Young's modulus and Poisson ratio) and the substrate curvature before and after deposition. However, it follows that effects of post-deposition processes on released structures cannot be considered. The wafer curvature can be evaluated using a profilometer [76].

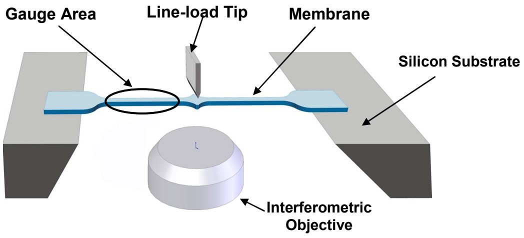

Fig. 9. MDE test.

Analytical extensions of Stoney's formula to discontinuous film and multilayers are also available in the literature [76].

### *3.3.6 Membrane deflection experiment (MDE)*

The MDE test was first reported in Ref. [77]. This test consists of transversely loading a membrane fixed at both ends and spanning a micromachined window beneath (Fig. 9). The membrane has a double dog-bone shape such that bending effects at the ends and where the load is applied do not control failure (Fig. 9). The transverse loading results in in-plane membrane tension of the microfabricated specimen.

The test uses the high load and displacement resolution of a nanoindenter with a wedge diamond tip. The real-time membrane deflection is recorded by a Mirau interferometer and a CCD camera [78] or by the out-of-plane ESPI (electronic speckle pattern interferometric) technique [79]. Hence, all the features of tensile methods apply to this test.

It has been used to measure fracture toughness in thin films [80] and to identify properties of nanomaterials, such as nanocrystalline diamond, amorphous diamond, and single crystal SiC films [81, 82].

### *3.3.7 On-chip bending testing devices*

Recently, a variety of bending based on-chip testing devices with integrated specimen has been presented. In Ref. [68] three different devices are described. The first two devices can load thin polysilicon specimens up to rupture by bending in the plane parallel or orthogonal to the substrate. Rotational and parallel plate electrostatic actuators have been used, respectively. The third device induces initiation and propagation of a crack in thick polysilicon specimen by a large number of comb-finger electrostatic actuators, which deform a notched specimen through a lever system. With these devices it is possible to determine the Young's modulus and the maximum stress at rupture of various materials.

## *3.4 Torsional test*

A special setup for carrying out pure torsional tests on small specimens was presented in Ref. [83]. Such a setup allows for measurement of torque and rotation undergone by a specimen with rectangular cross-section during the test. From these experimental results, the shear moduli of isotropic or anisotropic materials were obtained by means of two different inverse procedures, involving mixed analytical and numerical techniques. To the authors' knowledge this test has not been extensively used due to challenges in its implementation. Furthermore, torsional tests were also performed to investigate plasticity size effects on microwires [84, 85].

In the literature, it is also possible to find an example of an on-chip torsional test [44], which was performed to evaluate the maximum shear stress of a single crystal silicon bar. However, the proposed result is affected by high uncertainty, due to the uncertainty on some calibration parameters.

### *3.5 Nanoindentation test*

Instrumented micro- and nano-indentation techniques have been developed between the 1970s and the 1980s [86-88], but they have become very popular since 1992, when Oliver and Pharr [89] introduced an improved analytical method to correctly determine hardness and elastic modulus from nanoindentation load-penetration depth curves. Thousands of papers on this topic have been published and a number of reviews devoted to nanoindentation are available in the literature [90-93]. Such a method is widely used and, even if it was initially developed for studying bulk materials, many researchers have adapted it to investigate thin films. The method requires some preliminary knowledge of the specimen's behavior, which must be assumed or identified using inverse methods. In this regard, nanoindentation is much less accurate than tension or compression tests in characterizing material behavior. Its popularity is based on the sample preparation simplicity, which is countered by the complex data reduction, which typically involves numerical models. Furthermore, the presence of the substrate supporting the specimen may become a source of errors (substrate effect), which has to be taken into account for a correct analysis [94]. Finally, such a technique cannot be obviously applied to study the behavior of 1D nanostructures, like nanotubes or nanowires.

The experimental load-penetration depth curves contain useful information to derive the hardness [95], the residual strains [96], Young's modulus [97], and the toughness of the sample [98]. Indentation measurements have been applied to a wide range of materials, ranging from metals, ceramics, polymers, and composites.

## 4. Dynamic tests

### *4.1 Fatigue tests*

Small-scale components often have to withstand cyclic loads during their service life. Free-standing films, usually used to fabricate functional and/or structural components in MEMS, are subjected to mechanical fatigue loading at different frequency in different kinds of environments. Thin films on a substrate, widely used for fabricating microelectronic devices and integrated circuits, are subjected to temperature variation during service unavoidably leading to fatigue damage. In any case, fatigue causes continuous change in the properties of the material, which can be a cause of a possible failure mechanism. A knowledge of the dynamic properties of materials is a key issue for correct engineering design.

During the last decades, many efforts have been made in order to develop fatigue tests suitable for micro- and nanoscale investigation. Fatigue tests are performed to characterize fatigue behavior of small-scale samples in terms of stress versus number of cycles to failure (S-N curves), fatigue damage, and fatigue crack initiation and growth. Metal materials were widely investigated (a good review on fatigue of small-scale metal materials is reported in Ref. [99]), but also micrometer-sized specimens of both single-crystal and polycrystalline silicon have attracted attention [68].

Fatigue testing methods can be roughly grouped according to the load they require. This can be uniaxial cyclic or dynamic bending, even if in the past other testing modes were used [100].

The most common tests involve uniaxial cyclic loading. Both tension-tension and tension-compression modes were used. However, the latter condition is less common, because buckling can occur [101]. Apparatus for fatigue tests under tension-tension loading were developed for testing thin metal wires [101, 102], metal [103-107] or silicon [108] free-standing films or foils, while metal films deposited onto a substrate can be tested by tension and compression loading modes using the test method proposed in Ref. [109]. Tension-compression cyclic loading tests can also be carried out on thin free-standing films. In this case, micro-cantilever beams are excited by dynamic bending under constant load amplitude control provided by repeated indentation [110, 111]. Fatigue life was derived from the variation of the microbeam stiffness during the test. A similar bending based methodology was proposed in Ref. [112] for testing thin films deposited onto a substrate, under tension-compression loading. Recently, a dynamic bending method based on the resonant frequency method was proposed in Ref. [113]. This method allows for investigation of the fatigue damage behavior of metallic multilayers and/or thin films under very high frequency and to monitor the fatigue failure lifetime [114]. However, with this methodology, the fatigue failure is supposed to occur when a 10 Hz reduction of the sample's resonance frequency is measured, which corresponds to the development of cracks above a certain size.

A method for thermal fatigue testing of thin metal films and lines was proposed in Ref. [115]. Films bonded to a Si substrate are subjected to cyclic strain and stress due to temperature cycles generated by Joule heating due to an alternating current circulating on the thin metal specimen. With respect to bending and uniaxial cyclic loading, thermal loading has the advantage of not requiring mechanical actuators, but only a pair of electrical contacts. This means that this kind of test can be easily performed within a wide range of frequency (depending on the frequency of the applied AC voltage) and *in* *situ* electron microscopes. However, with this technique, only metals, and not freestanding samples, can be tested. Moreover, the main limitation is in the relationship between the temperature increase and the strain, which cannot be independently varied. In fact, the induced strain is a function of the temperature and the mismatch between the expansion coefficient of the sample and the substrate. Finally, stresses cannot be measured during the test. Therefore, experiments are performed under strain control and the corresponding stress value is derived from stress-strain curves independently determined from wafer curvature tests. This means that cyclic hardening or softening is neglected [115].

All of the aforementioned uniaxial and bending methods and those in Refs. [116-120] require the use of external stress or strain sensors for measuring the load applied by the actuation systems.

Another category of methods includes on-chip devices, which are usually actuated by comb-drive structures [48, 121] or piezoelectric elements [122]. Examples of tension-tension and compression-tension are reported in Refs. [48, 122], while examples of bending are reported in Ref. [121].

### *4.2 Resonant tests*

Resonant tests are non-destructive tests, that allow one to evaluate the elastic constants and/or the residual stresses by measuring the resonance frequencies of material samples in the shape of micro or nano beams [123-126], square or circular thin plate [127-128], and supported thin films [129].

In all these cases, analytical or semi-empirical expressions, which correlate the geometry, the mass, the stress field and the material properties of the sample to the resonant frequency values, are used. The simple value of the fundamental resonant frequency allows for the determination of Young's modulus or residual stresses if it is measured for, respectively, a cantilever beam [130-133] or a beam simply supported at both ends [134]. Simultaneous determination of both Young's modulus and residual stresses can be carried out in beams with clamped-clamped ends [124]. Two resonant frequencies of thin square or circular plates with free boundary condition are required for simultaneously determining Young's modulus and Poisson's ratio [127, 128]. Twenty or more free vibration resonance frequencies were required for determining the anisotropic elastic constants of thin films deposited on a substrate by the resonant ultrasound spectroscopy method (RUS) [129].

Uncertainties related to non-ideal supports and disturbing phenomena, like air damping and squeeze-film air damping, provide sources of error that can lead to significant shifts in the resonant frequencies of microfabricated structures.

Mechanical properties of micro-components can also be determined by the use of atomic force microscopy (AFM). In particular, one of its most important applications for the mechanical testing is called atomic force acoustic microscopy (AFAM), which is based on the observation that the resonant frequencies of the AFM cantilever change when the cantilever tip is put into contact with the sample surface [68]. An analytical model is used to relate these frequency data to the sample-cantilever tip coupling, which is usually modeled as a spring [135]. Then, from the stiffness of such a spring the local mechanical properties of the sample, like its Young's modulus [136-137], can be derived. Samples that can be tested by the means of the AFAM can be of different shape, such as thin films [138] or nanowires [139].

## 5. Research trends

About six hundred articles, published in international journals since 1920, have been consulted by the authors in order to analyze, summarize and trace the temporal evolution of the efforts by the scientific community in micro and nano-scale mechanical characterization of materials[2]. In this section, the results of such an investigation are summarized and discussed. In particular, the attention is focused on the mechanical quantities that can be determined by the test methodologies described in the previous sections, on the investigated materials and on the interest the researchers showed toward each specific material, and each test methodology.

Regarding the mechanical properties that can be determined by the aforementioned tests, a complete picture of the offered possibilities is given in Table 1 (where the symbol X confirms the suitability of a specific test to determine a specific quantity).

The pie charts reported in Figs. 10 and 11 show (in terms of percentage of the examined papers) the interest of researchers in the testing methods and the materials, respectively. The bar graph of Fig. 12 illustrates instead the temporal evolution of the interest toward different kind of materials.

As can be inferred from Fig. 10, tension is the test methodology most surveyed and used. This is not a surprise since, as it can be noted from Table 1, it is the most versatile among all the other test methodologies. In fact, it allows one to completely characterize the mechanical behavior of a material sample. By deriving its full stress-strain constitutive curve, mechanical properties like the Young's modulus and the ultimate stress, or information about the basic processes of plastic deformation or creep behavior can be determined. Moreover, this methodology can be used for both micro and nano applications and for testing a great number of materials (see Table 2).

It must be noted that, still today, there are neither standardized methodologies nor unified test equipment for micro or nano tension tests. Every researcher is free to develop and use his own test apparatus, and the convergence on a definitive and universal method has not occurred. The most interesting promise comes from the generation of on-chip stages. These systems allow *in situ* tests and reduce the alignment troubles, which are the major sources of error in tensile tests, by co-fabricating actuators, sensors, and sometime the specimen in a unique chip. Furthermore, since such stages are electromechanical systems, they could provide combined electrical and

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[2] For the sake of brevity, only a limited number of these papers are mentioned in the references.

Table 1. Mechanical tests and determinable properties.

| TEST | | | IDENTIFIABLE QUANTITY | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | Young's modulus | Poisson's ratio | Shear modulus | Yield strength | Fracture strength | Creep | Residual stress |
| Tension | | | X | - | - | X | X | X | - |
| Compression | | | X | - | - | X | - | - | - |
| Bending | Axisymmetric | | X | - | - | - | X | - | - |
| | Bulge | | X | X | - | X | X | - | X |
| | MDE | | X | - | - | X | X* | - | X |
| | Microbeam | | X | - | - | - | X | - | X |
| | M-test | | X | - | - | - | - | - | X |
| | Wafer curvature | | - | - | - | - | - | - | X |
| Torsion | | | - | - | X | - | - | - | - |
| Nanoindentation | | | X | - | | - | - | X | X |
| Resonant | Sonic | beams | X | - | - | - | - | - | X |
| | | plates or fims | X | X | - | - | - | - | - |
| | RUS | | X | X | - | - | - | - | - |
| | AFAM | | X | | | - | - | - | - |

| | | S-N curves | Fatigue damage | Crack growth rate |
|---|---|---|---|---|
| Fatigue | Uniaxial tension | X | X | X |
| | Bending | X | X | X |
| | Thermal | X | X | X |

*Can also identify fracture toughness

mechanical tests on samples. On the other hand, they require quite an accurate and careful design.

Bending test methods come after tensile tests in importance (Fig. 10). These test methods are relatively easier to apply compared to tensile tests, but in general, they allow one to determine a reduced number of micro and nano-scale properties (see Table 1). Among the variety of bending tests, the bulge test is the most flexible, providing the possibility of determining properties such as Young's modulus, Poisson ratio, the yield and fracture strengths, and also residual stresses, and it was used to investigate many materials (see Table 2). The MDE and microbeam tests have been successful, and can be inferred from Table 2, a good number of materials has been studied. The axisymmetric test, the M-test, and the wafer curvature tests have been used less, probably due to the limited information and accuracy they can provide. As for tension tests, on-chip stages seem to represent a valid choice for determining elastic properties and strength of various materials.

Dynamic tests have been extensively used (Fig. 10). Uniaxial tests are the most common configurations within the fatigue tests. They have been applied for testing metallic and ceramic micro and nano-scale samples (see Table 2) with the aim of determining the variation of the stress versus number of cycles to failure (S-N curves), fatigue damage, and fatigue crack initiation and growth. Resonant test methods prevalent as an alternative to static tests for determining the Young modulus and residual stress. With particular reference to the residual stress, resonant tests can be preferred over bending wafer tests, like the wafer curvature method, since they can consider the effect of post-deposition processes on released structures [131].

The remaining test methods such as compression, fracture mechanics, torsion test, Raman spectroscopy [140, 141] for residual stress measurement, hole drilling method [142] for measuring the isotropic elastic constants, Brillouin scattering techniques [143] for measuring anisotropic elastic constants, etc., have been less frequently applied because of their complexity or the limited number of information they can provide.

It is worth pointing out that today the nanoindentation test is the most used and diffused test for measuring hardness and Young's modulus, likely due to the simplicity in sample preparation and the availability of commercial equipment. Nanoindentation is also applicable to investigations of residual strains and toughness. A very high number of papers were published on this topic, and by a rough analysis carried out by the authors they represent more than one third of the total number of the papers examined under this review. Indentation measurements have been applied to a wide range of materials, ranging from metals, ceramics, polymers to composites. Despite these facts, for the sake of clarity and because of the important role played by the other techniques, in particular the

Table 2. Materials investigated with the main testing methodologies.

| | | MATERIAL | | | | LENGTH SCALE | |
|---|---|---|---|---|---|---|---|
| **TEST** | | Ceramic (silicides) | Metals | Polymers | Others | Micro | Nano |
| Tension | | Si[28, 146]<br>SiC[148]<br>SiN[149]<br>$SiO_2$[147] | Au[144, 147]<br>Al[145]<br>Cu[159]<br>Ag[30]<br>TiN[35] | Epoclad[16]<br>Epoxy[16]<br>PAN[190] | SiGe [150]<br>CNT [151]<br>ZnO [152]<br>DNA[191]<br>GaN [192] | X | X |
| Compression | | Si[158] | Mg [156]<br>Au [57,154]<br>Mo [155]<br>TiAl[157]<br>Ni [56,153] | - | - | X | X |
| Bending | Axisymmetric | Si[61, 62] | - | - | - | X | - |
| | Bulge | Si[165]<br>SiN [160] | Au[166]<br>Al [166]<br>W[166]<br>Cu [70] | - | - | X | X |
| | MDE | SiC[81] | Au[78, 170] Al[167, 169]<br>Cu[167, 169] | - | Nanocrystalline diamond[81,168]<br>Amorphous- tetahedra diamond[81] | X | - |
| | Microbeam | Si[63]<br>SiN[65] | Cu [162]<br>Ni [164]<br>Au[173] | - | CNT[163]<br>ZnO[188]<br>GaN[193] | X | X |
| | M-test | Si[72] | Au [73] | - | - | X | - |
| | Wafer curvature | SiC [172] | Ni[171]<br>Al[171] | - | - | X | X |
| Torsion | | Si[83] | Ni [83]<br>NiFe [83]<br>Cu[84, 84] | - | - | X | - |
| Nanoindentation | | Si[98]<br>SiC [172] | W [161]<br>TiNi [173]<br>Al [89]<br>Steel[179]<br>Cu[180]<br>TiN[181] | [175] | SiGe[150]<br>C [182] | X | X |
| Resonant | Microbeam | Si[125]<br>SiC [132] | Au [131]<br>Ni [123] | Polymide [134] | GaN [176]<br>ZnO[126,133] | X | X |
| | Thin film | Si[129] | Cu [127] | | CVD[127,128] | X | - |
| | AFAM | Si[177] | Ni[178] | - | - | X | X |
| Fatigue | Uniaxial | Si[108,116,117] | Cu[119,120,183]<br>Al[122]<br>Ni[118]<br>NiCo[187] | - | Collagen [48] | X | X |
| | Bending | Si[121, 185, 186]<br>SiN[184] | Steel [111] | - | - | X | - |
| | Thermal | - | Cu[115] | - | - | X | - |

tension and bending tests, nanoindentation was not considered in the diagrams of Figs. 10, 11 and 12.

As previously shown, the literature includes a variety of tests to investigate material properties at small length scales, and each of them offers different opportunities of study and requires more or less sophisticated experimental setup. So, the choice of one methodology instead of the others depends on the scope of the researcher and the available instrumentation. Surely, tension tests have great potentiality, especially in the form of on-chip test devices, since they allow one to determine a wide variety of materials' properties. However, in light of the complicated apparatus they require, a good alternative to them can be bending tests. However, they give less opportunity. For studies focused on plasticity, we suggest compression tests.

With regard to the most studied materials, it results from Table 2 that a variety of metallic, ceramic and polymeric materials were tested: silicon, silicides, copper, nickel, and aluminum are the most common. The pie chart of Fig. 11 shows the interest for each specific material.

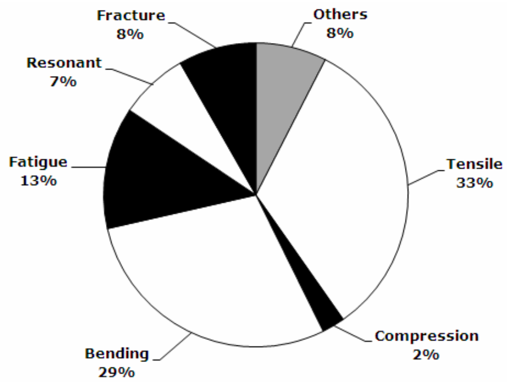

Fig. 10. Test methods used to characterize micro- and nanosamples.

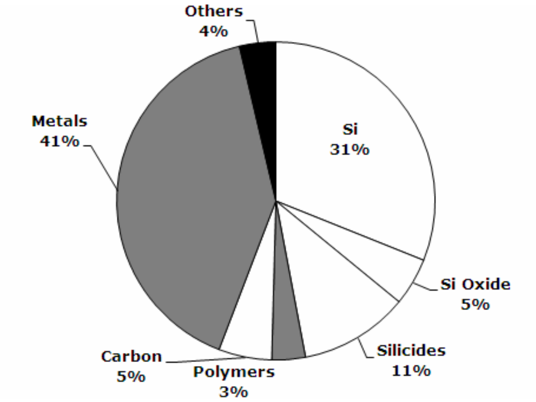

Fig. 11. Materials tested at micro and nano-scale.

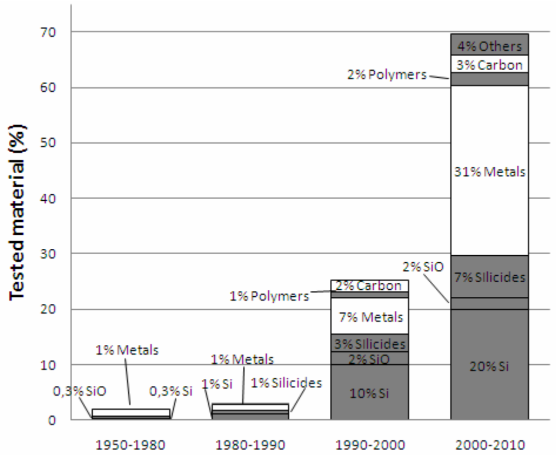

Fig. 12. Materials tested at micro and nano-scales between 1950 and 2010.

It can be observed that silicon (single or polycrystalline) and its compounds (silicon oxide and silicides, such as silicon carbide and silicon nitride) have been the most intensively studied materials. Great consideration has also been given to metals. Carbon nanotubes, polymers and other materials (as GaN and ZnO nano wires) follow in decreasing order.

Finally, the bar graph reported in Fig. 12 highlights the importance given to each material from 1950 to 2010. It can be verified that the interest on silicon (the first and the most important material used by the microelectronics industry since the 1990s) and its compounds has almost always surpassed the interest for other materials. However, the importance of metals inside MEMS is increasing and leading toward a renewed interest in their characterization at the microscale. Interest in nanocrystalline diamond, silicon carbide (SiC), and amorphous diamond as new MEMS materials is also growing.

Moreover, from the graph it is possible to appreciate the growing interest in carbon nanotubes, which could represent an excellent alternative to traditional materials thanks to their highly improved electrical, photonic, and thermal properties as compared with their bulk counterparts.

## 6. Concluding remarks

In the present paper, we reviewed the most relevant literature on mechanical testing of materials at small length scales.

The most important test methodologies were identified, classified and briefly described. So far, tensile and bending tests have been the most commonly adopted static tests (other than nanoindentation) and, among these, on-chip stages seem to be the most promising. The increasing interest on the fatigue tests for studying the behavior of both metallic and silicon-based materials was also pointed out.

Attention was paid to the most investigated materials and the mechanical quantities that can be determined by each test method. The characterization of metals, silicon carbide, and diamond materials is attracting growing interest. In fact, even if they are widely used in microelectronics and MEMS, there is still a lack of information about their properties. As regards to the mechanical quantities that can be determined by each test method, it must be observed that while all the available methods are suitable for determining Young's modulus, only a few provide Poisson's ratio. Moreover, bending and nanoindentation tests are the most suitable for residual stress measurement. The compression test is mainly used to study plasticity while only tension tests allow one to study creep behaviour of materials.

Despite the great work carried out for developing more accurate, reliable and simple methodologies, at present, there are still neither standard methods nor universally recognized fixtures for mechanical testing of materials at micro and nano-scales. Many solutions have been presented over time, but still today new methodologies are required and emerging to overcome limits of the existing ones. New testing methodologies are also needed to support the continuous advance of micro and nanoelectronics, as well as biology, for a deeper understanding of the behavior of materials like nanofibers, nanotubes, nanostructured, and biological samples. However, a good starting point is the study of the developed methods included in the literature. Therefore, a large number of appropriate references are selected and proposed in this paper to allow the interested reader to find detailed information concerning materials and their properties, and above all the methodologies used by the researchers for testing them. All the results of the bibliographic investigation carried out by the authors are summarized in table or graphic format for easy consultation. We hope this review serves as an introduction to newcomers to the field of nano/micro scale testing.

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