STATE OF ART IN MICRO- AND NANO-SCALE MECHANICAL CHARACTERIZATION OF MATERIALS

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Introduction

Micro-electromechanical systems (MEMS) are now widely used in a variety of industrial, biomedical and consumer applications. Their excellent qualities, as small size, high performances and low-power consumption, have allowed them to get an extraordinary success during the last 20 years. Such a success has driven the interest of the scientific community toward the understanding of materials behavior at micro and nano-scale.

It is now well known that materials with submillimeter dimensions do not behave as expected at macroscopic dimensions. For this reason, mechanical properties of such materials cannot be derived from test performed on ordinary macroscopic specimens. Moreover, standard machineries commonly used for macroscopic mechanical testing are not suitable to manipulate micro- and nano-samples. So, many new and appropriate methods have been developed in order to investigate elastic properties, fracture limits, residual stresses, dynamic properties and so on. Starting from some excellent review articles [1-6] and considering several other papers and books not mentioned herein for the sake of brevity, the present investigation attempts to update the current state of the art in the field. About four hundred papers, published on international journals from 1920 up to now, have been consulted in order to trace the temporal evolution of the efforts involved by the scientific community in micro and nano-scale mechanical characterization of materials. In particular, the materials and the corresponding mechanical properties, the specimen sizes and the test methods more commonly studied have been identified and analyzed.

Experiments for micro-and nano-scale testing

The accurate measurement of the mechanical properties of thin films and nanoscale structures, such as nanowires, nanorods, nanotubes, and nanobelts of various materials has led to the development of several different test methods. Attention was focused on test methods estimating properties related to the elasticity, the strength and the fatigue behaviour of the material. In particular, properties such as Young's modulus, Poisson's ratio, the fracture strength, the fracture toughness, the residual stress field and the response to cyclic loading in terms of the *S–N curve*, related to the number of cycles at rupture have been investigated. These methods mainly include quasi-static tests, as compression, tension tests, bending tests (including axisymmetric bending, microbeam bending, bulge test, M-test, and wafer curvature test), and dynamic methods, like resonant and fatigue tests.

As regards the materials, a variety of materials such as Silicon, Silicon oxide, silicides (SiN and SiC), metals (as gold, silver, copper, aluminum), polymers, carbon nanotubes and nanowires have been studied.

Analysis of the data and discussion

Almost all research activities on the mechanical characterization of materials at micro- and nano-scale have been carried out in the last six decades. Figure 1 shows the trend of scientific production (percentage of published works versus time). Up to 1980, only few papers were published whereas there is a close to exponential increase afterwards. The scientific activities begin to increase towards the 1980s, concurrently with the production of the first MEMS inside some research laboratories. From the 1990s, the great market success of MEMS has been a further stimulus for the scientific community giving as a result a strong increase of production. More than the 65% of the activities have been carried out in the last ten years.

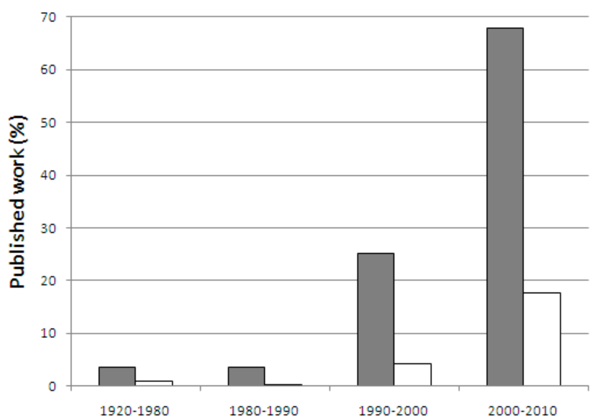

Figure 1 Trend of the published production: in gray the global production; in white the production related to nanometric specimens.

In the same graph of Fig.1, the percentages of works involving the nano-scale are also reported. It is worth to note that in the last years the interest for nano-scale has grown up as well as the interest for micro-scale even if this last remains always higher. The reason for such a trend is related to the ever-increasing interest toward very promising nanostructures, like carbon nanotubes and nanofibres.

The pie charts of figure 2 and 3 show the percentage of the examined papers in which a specific test method was proposed and/or used and the percentage of papers in which a specific material was examined, respectively.

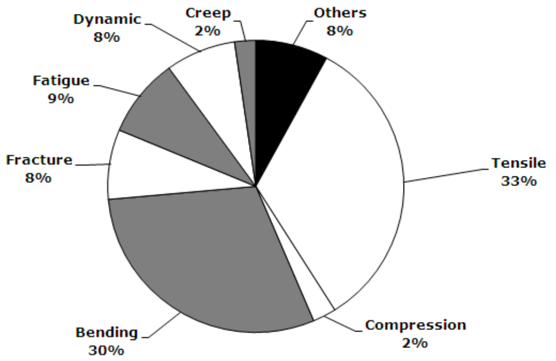

Figure 2 Test methods used to characterize micro- and nanosamples.

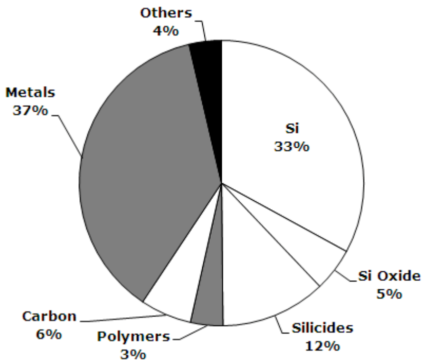

Figure 3 Materials tested at micro and nano-scale.

As can be inferred from figure 2, tension/compression tests have been the more commonly used. Properties such as Young's modulus, Poisson's ratio, yield strength and fracture strength can be directly determined with these tests. Bending test methods follow straight after. These test methods are relatively easier to apply and allow the indirect measurement of micro and nano-scale properties such as Young's modulus, residual stresses and fracture strength. A series of other test methods such as creep test, fatigue test, fracture mechanics test, dynamic test and others (Raman spectroscopy, hole drilling method, torsion, etc.) have been applied with a reduced intensity.
As regards the materials, which were examined, it can be observed from figure 3 that silicon (single or polycrystalline) and its compounds (silicon oxide and silicides, such as silicon carbide and silicone nitride) were surely the more intensively studied material. Great consideration was also given to metals and carbon nanotubes, polymers and others (as GaN and ZnO nano fibers) follow in decreasing order.
The bar graph reported in figure 4 highlights the importance given to each material during the time. It can be verified as the interest toward silicon (the first and the most important material used by the microelectronics industry since the 1990s) and its compounds has been always overcoming the interest for other materials. However, the importance of metals inside MEMS is increasing and leading toward a renewed interest about their characterization at microscale.

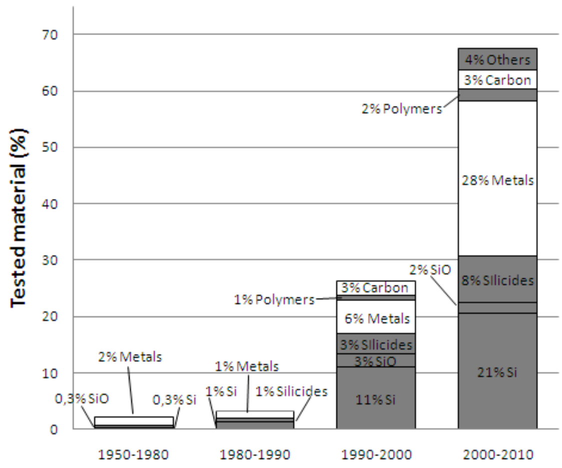

Figure 4 Materials tested at micro and nano-scale.

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

Concluding remarks

From the previous analysis, it is possible to highlight that: 1) The studies about nano-samples are increasing, but not as those about micro-samples. This can be explained since systems using nano-components are not so market available as MEMS; 2) Characterization of metals is attracting growing interest. In fact, they are widely used in microelectronics, but there is still a lack of information about their properties, because, during the past, attention was mainly put on Silicon; 3) Most of the test methodologies deal with quasi-static properties of materials, while less data are available for their dynamic behavior.

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