ADVANCING TECHNOLOGY IN MEXICO – CHALLENGES FOR NANOMETROLOGY

ZAAWANSOWANE TECHNOLOGIE W MEKSYKU – WYZWANIA DLA NANOMETROLOGII

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Abstract: Products based on nanostructured materials are already present in our daily lives and have gradually become more important in Mexico. Nanomaterials can be found in many applications, such as catalytic processes, automotive parts, coatings, electronics, etc. The diversity and complexity of manufactured nanomaterials are continuously increasing as well as their use in industrial process and consumer products. It has been long recognized that nanostructured materials can have significantly different properties when compared to their larger scale counterparts. Measurements are essential to have a complete understanding of any new phenomenon or process in the nanoscale. There are numerous properties and quantities to be measured in the nanomaterials used in industrial processes and the establishment of metrological traceability to the SI units is a challenge to deal with.
Techniques, methods, measurement standards and infrastructure development are needed to control manufacturing and production, to ensure product quality and enable different elements to work effectively together. This paper describes issues and challenges in metrology to be faced in Mexico concerning nanotechnology.
Keywords: metrology, nanometrology, measurements in nanoscale, nanomaterials

Streszczenie: Produkty na bazie materiałów nanostrukturalnych są obecne w naszym codziennym życiu i stopniowo stają się coraz ważniejsze w Meksyku. Nanomateriały stosowane są m.in. w procesach katalitycznych, częściach samochodowych, powłokach, elektronice itp. Różnorodność i złożoność produkowanych nanomateriałów stale wzrasta, podobnie jak ich zastosowanie w procesach przemysłowych i produktach konsumpcyjnych. Od dawna uznaje się, że materiały nanostrukturalne mogą mieć znacząco różne właściwości w porównaniu do swoich odpowiedników w większej skali. Pomiary są niezbędne, aby mieć pełną wiedzę o wszelkich nowych zjawiskach i procesach zachodzących w nanoskali. Istnieje wiele właściwości i wielkości do zmierzenia w nanomateriałach stosowanych w procesach przemysłowych, dlatego wyzwaniem jest zapewnienie identyfikowalności metrologicznej

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jednostek układu SI.
Potrzebne są techniki, metody, normy pomiarowe oraz rozwój infrastruktury do kontroli wytwarzania i produkcji w celu zapewnienia jakości produktu oraz umożliwienia skutecznej współpracy różnych elementów. W artykule opisano problemy i wyzwania metrologii w zakresie nanotechnologii, które napotkano w Meksyku.
Słowa kluczowe: metrologia, nanometrologia, pomiary w nanoskali, nanomateriały

1. INTRODUCTION

Over the past decades, manufacturing has experienced significant changes as rapid globalization shifted a substantial proportion of manufacturing capacity from developed to emerging economies and substantial new markets and new competitors emerged. The globalization of manufacturing was enabled by a combination of strengths coming together simultaneously, including a significant change in the geopolitical relations among countries, the widespread growth of digital information, physical and financial infrastructure, computerized manufacturing technologies, and the proliferation of bilateral and multilateral trade agreements [1].

The manufacturing industry is, and has been of great interest to investors and business executives aiming to take advantage of the opportunities presented by globalization and the significant growth of emerging markets, as well as serving to high-value customers in developed markets with innovative new products and services. Four strategic sectors have been identified for economic growth and development of Mexico. These sectors are automotive, electronics, aerospace and medical devices. Mexico is well positioned to export manufactured goods due to its 11 free trade agreements with 43 countries, allowing both domestic and foreign manufacturers to have free-trade access to more countries than any other in the world [2]. Manufacturing will benefit from important innovations in materials, product design, production process, and manufacturing business models. Advances in lightweight materials, additive manufacturing, thrifty innovation, and recovering and recycling materials used in finished products, will change how manufacturers use metals and other materials and raise resource productivity and efficiency. The need for new capabilities and higher performance in materials, the need for bigger customization, and a greater focus on long-term cost and resource sustainability are all driving innovations in materials. Nanotechnology is enabling these advances in manufacturing industries as varied as automobile and medical. The main challenge is to develop seamless integration of technologies and processing for using nanomaterials in production; to improve the control and monitoring of the conditions required for the use of nanomaterials in industrial processes, including metrology; to increase the level of robustness and repeatability of such industrial processes; to optimise and evaluate the increased performance and functionality of the product and of the production line.

Nanotechnology is nowadays a worldwide issue with a scientific relevance due to its impact in trade, health, environment and safety. This emerging technology has had a relevant development in Mexico along the last decade. The number of industries using nanotechnology in their process and research publications reflects this growth. There is a preliminary inventory of 101 companies from different manufacturing sectors [3] including those above mentioned (Fig. 1). Several of these industries are gathering as a model of industrial cluster. This model is considered a suitable mechanism to promote development, meaning the stimulus of innovation for regionally economic growth [4]. Most of this industries use nanomaterials in their process and only a small number produce them.

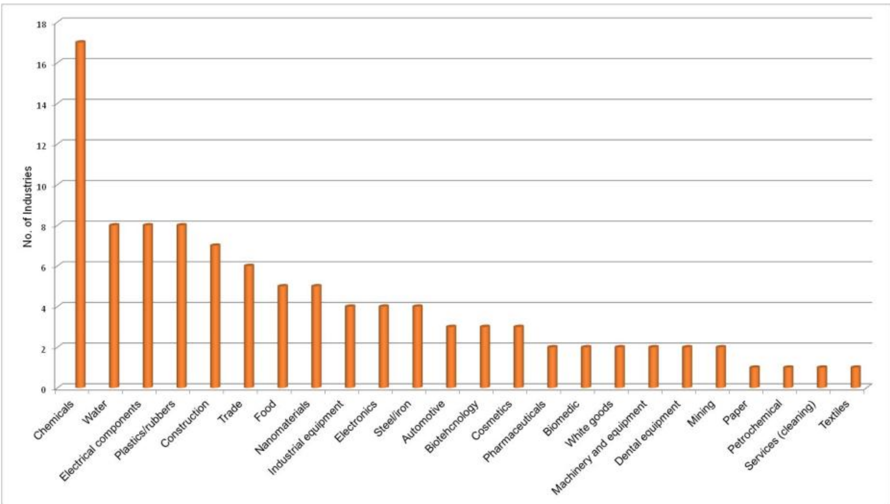

Figure 1. Distribution of industries using nanotechnology in Mexico [3]

Nanomaterials frequently are designed to exhibit new properties as compared to their bulk counterparts. Nanostructures are of large importance to create materials with new properties and functions to be exploited in many different areas. The use of nanomaterials in manufacturing is still relatively new and limited in Mexico, and a number of challenges stand in the way of increased use. Metrology has to be seen as an essential part of nanotechnologies and it is a one of this challenges. Any scientific activity leading to technology applications must be accompanied by reliable measurements.

Entirely new metrology tools and analysis methods are required to meet the needs of the emerging nanotechnology industry. While much progress has been made, current instrumentation and metrology are at their limits and much greater capabilities will be required, from laboratory to commercial scale manufacturing as well as from the National Metrology Institutes, the Centro Nacional de Metrologia (CENAM) for Mexico.

2. CHALLENGES FOR NANOSCALE METROLOGY

Measurement is ever-present and very much taken for established in the modern developed world. Accurate measurements are required for a diversity of items, since the accuracy of electrical power metering to ensure the bill of energy we have to pay through having the correct dose of radiotherapy for cancer treatment. Good measurement allows countries to remain competitive, trade throughout the world on an equal footing and, hence, improve the overall quality of life.

CENAM mission is focused on this effort as the national metrology institute of Mexico, and so it is working on a program for establishing a nanometrology infrastructure to support

national measurement needs. This infrastructure is aimed to link practical measurements made in manufacturing industries to the international system of units (SI), embodied in the Mexican national measurement standards, through a hierarchy of measurement standards, traceable to internationally recognized measurement standards and SI units at the end. The infrastructure ensures that measurements can be made on a consistent basis throughout the country and in-line with international measurement practices [5].
The great challenge for a reliable production is to develop the capability to reliably and reproducibly image and measure any nanostructure for any relevant property with atomic accuracy in three dimensions. Realizing the potential of the emerging nanotechnology industry in Mexico will require high performance, cost-effectiveness, reliable instrumentation; improved measurement methods; information and data transmission and interpretation; globally-accepted standards for measurement; identification of properties and structures at the nanoscale in all life-cycle of products as shown in Figure 2.

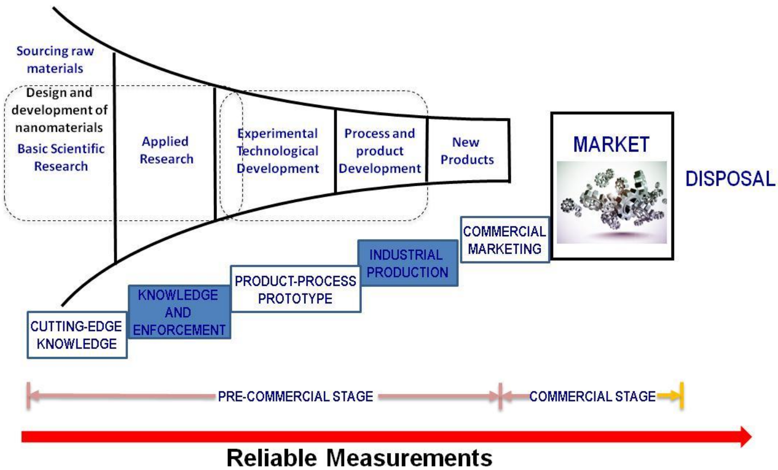

Figure 2. Reliable measurements are required in the full life-cycle of nanotechnological products [6]

The main difference between nanotechnology and many other fields of science or engineering is the size. Dimensional metrology, which has its roots in engineering and physics, needs to meet the challenges of expanding into this emerging technology and it may be involved in the whole life-cycle of products. The wide scale use of precision engineered, improved functionality devices and components is dependent on the future provision of traceable dimensional measurements at the nanoscale. An important measurement instrument in this area is the scanning probe microscopy (SPM) that is currently mainly used to give qualitative information. There are still several technological barriers to overcome before rapid quantitative information can be achieved as calibration and metrological traceability.
Accurate measurement of dimensions and elucidation of structures are critical to the development of nanomaterials and devices. With the increasing importance of nanomaterials in fundamental research, technological and manufacturing applications in Mexico, it is important consider the nature of these often unexpected challenges associated with reproducible characterization of nanomaterials, including the difficulties of maintaining

desired materials properties during handling and processing due to their dynamic nature. It is equally critical to understand how characterization approaches can provide reliable information and to determine how materials and properties change in different environments. The establishment of metrological traceability on nanomaterials characterization is a real challenge.

2.1. CHALLENGES FOR CHARACTERIZATION OF NANOMATERIALS

Characterization of nanomaterials cover issues in physical and chemical metrology, including force and length measurements, chemical composition determination, shapes of pores and particles, and 3D relationships of complex nanoscale components. A combination of measurement capabilities will be needed to address characterization challenges at the nanoscale. These either will extend existing measurement techniques such as imaging by microscopy and analysis by spectroscopy, or will emerge from the invention of new measurement methods that enable both compositional and performance factors to be quantitatively and reproducibly measured on the nanoscale.

Nanoparticles are one class of materials that is very important for nanotechnology products.The properties of particles depend extremely on their size, for example in quantum dots, where the size-dependent degree of exciton confinement determines the wavelength of the photoluminiscent emission [7]. Materials that are considered inert in their bulk form can exhibit catalytic behavior at the nanometer-scale due to the increase on the surface area, which opens an avenue to potential industrial applications of these materials but also prompts important environmental, health, and safety considerations.

An important barrier that hamper both application and health and safety issues are reliable large scale production and in particular accurate assessment of the nanoparticle characteristics such as size, shape, state of agglomeration and surface chemistry. The importance of the assessment of particles size for many applications at the nanoscale is of great interest but not always quick and easy and when the size distribution is important and not just a comparative median needed then the task is even less straightforward. For instance, the production of ceramics from nanosized powders the particle size distribution and state of agglomeration both are essential characteristics. Agglomeration can have a huge influence on particle packing, sintering and resulting microstructure, grain size and properties [8].

There are numerous techniques used to determine the dimensional properties of nanoparticles [9]. However, they are more or less well suited to particles in different size ranges and size dispersions. Several international efforts had been made in order to obtain validated and traceable size determination results of nanoparticles [10,11] where CENAM also participated but there are still several issues to be addressed using validated methods and procedures that can be used for all laboratories. New reference materials with certified size, shape, composition and materials properties need to be made generally available to verify these new procedures and required to allow comparison between techniques. It is also necessary to estimate the measurement uncertainty.

2.2. NEW MEASUREMENT STANDARDS

Nanotechnologies require a big number of quantities to measure. Nevertheless, among quantities like surface area and chemical composition, dimensions are often considered as the flag to enter into the nanoscale, the size range from approximately 1 nm and 100 nm [12]. It is noted that the nanoscale features the nanotechnologies. It is also worth to note that some tolerances in manufacturing have reached, or at least have got close to the upper part of the nanoscale, namely 0.1 μm. The coatings and films thicknesses are often in the nanoscale as well.

The manufacture industry specially requires dimensional good agreements along the chain value where several suppliers are usually contributing. Interchangeability continues as a vital feature nowadays. Manufacturing needs equivalent and reliable dimensional measurements, which have been accomplished fairly well in the industry along, now we could say, several centuries. The manufactured products available today constitute the demonstration of this success. So, the measurements done in the floor are reliably related to measurement standards within the plant, which are related as well to other measurement standards in other laboratories, to reach those under the responsibility of the national metrology institutes (NMIs) in each country, which are finally related to the definition of the agreed SI measurement unit for length: “The metre is the length of the path travelled by light in vacuum during a time interval of 1/299 792 458 of a second” [13]. It is said that this property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations is called metrological traceability [14]. The NMIs are responsible to provide the references for metrological traceability and to ensure that these references are equivalent to those taken in other countries, even by means of formal recognition agreements [15].

Of course the characteristics of these chains have evolved to take into account the ever increasing demands for major accuracy and precision from the industrial advancements. Also, the length ranges have changed, to extend up to geodesic length measurements and down to the sub-micrometer region. However there are still challenges to surmount within the nanoscale, seemingly due to the requirement is relatively new and that not enough developments in the measurement technology are in place. The necessary method validation and comparisons among laboratories are still in process. Thus, it is not possible yet to achieve metrological traceability of the nanoscale measurements to the SI unit for length; it can be said that nowadays there is no certainty to strictly use the term nanometer as the hundred-millionth of the SI unit for length in the dimensional measurements at the nanoscale.

Artificial gratings are used as a step to go down the length scale towards the nanometer region. They are periodically spaced collections of identical features [16] and the spacing may be in the micrometer range, they can be characterized in length by optical diffraction to become measurement standards. Unfortunately the method using visible frequencies is limited in the lower range to approximately 200 nm, one half of the wavelength of visible light. One way to go further down is through electron and probe microscopy techniques. Nevertheless, there is not agreement among their results at measuring nanoparticle size [17] essentially due to technique-dependent differences on the measurand, the quantity intended to be measured [14].

The atomic force microscopy - AFM - , one of the probe microscopy techniques, has been quite useful because of the clarity of its application to the measurand. The AFM is a “method for imaging surfaces by mechanically scanning their surface contours, in which the deflection of a sharp tip sensing the surface forces, mounted on a compliant cantilever, is monitored” [18]. Thus, save corrections due to the dimensions of the tip and the like, the AFM results in a quite approximation to the particle or nano-object size, size distribution and shape. A nano-object is a material with one, two or three dimensions in the nanoscale [12]. A nanoparticle is a particular case of a nano-object whose all three dimensions are in the nanoscale. Although the AFM provides a great sensitivity to detect topographic minute details, it requires measurement standards to achieve measurement results metrologically traceable. These standards include gratings or devices containing structures at the nanoscale whose dimensional values are traceable. Some NMIs, like the PTB in Germany, have developed some of such devices.

CENAM is starting a project to establish a metrological link of the dimensional measurements at the nanoscale to the realization of the metre. The general idea is to calibrate a 3-coordinate laser interferometer with the stabilized laser that realizes the metre, and then attach it to an AFM to measure the tip displacements. This tool, called also a metrological AFM, will allow the dimensional characterization of devices to use them as dimensional measurement standards, and to apply them for calibration of commercial AFM, and others.

Another part of the project is aimed to build prototypes for such devices, so a virtuous cycle could be accomplished. This part of the project requires a microscope with an ion column.

The project outcome is expected to impact the national quality infrastructure to address, at less partially, the responsible and sustainable development of nanotechnology. It will provide reliable support either for the national industry to increase its competitiveness, by demonstration of their claims, as well as for the regulators to really implement the mechanisms to protect the human health and the environment.

3. CONCLUSIONS

Advances in nanotechnology require long time horizons and continued investments in materials, platforms, and applications across manufacturing industries. Further research is also needed to gauge the long-term environmental and health effects of products manufactured with nanotechnology.

Developments in nanotechnology place special demands on the required metrology infrastructure at the nanoscale, while at the same time, the rate of nanotechnology development continues to increase in both volume and complexity. Needs are more extensive than can be provided by individual actors, simply because all stages of the innovation process have to be covered at the same time as nanotechnology is rapidly developing.

A strengthening is needed of the existing network of Mexican measurement facilities as well as investments in the development and introduction of new standards and measurement techniques, as part of the necessary infrastructure to support growth in nanotechnology. Few such standards and techniques exist today which can be used across the nanoscale range and existing measurement techniques and instrumentation will have to be developed further as whole new ranges of nanomanufactured products will appear. Major research and development efforts have to be undertaken in order to respond to these challenges.

CENAM will have to encourage synergies between research institutions, government and the industry to face metrology challenges of manufacturing industries using nanotechnology.

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