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Surface technology

Precision work: defect-free coatings for silicon spheres

The prototype kilogram – to which all scales are calibrated to – is losing weight. International efforts are striving to redefine the base unit for measuring mass and, in future, redefine the kilogram on natural constants. For this purpose, the Avogadro experiment determines the number of atoms in almost perfect silicon spheres. Fraunhofer researchers have recently succeeded in homogeneously coating the spherical surfaces – the measurement uncertainty can be limited to a range below 10 micrograms.

Due to the fact that the prototype kilogram – the unit of mass upon which the weight of a kilo is based – is becoming increasingly lighter. The reason for this, however, is unknown. Now researchers are seeking alternatives for the platinum-iridium alloy artefact that is stored in a safe in Paris. The basic plan is to redefine the kilogram. In future, a physical constant will replace the material kilogram.

Thus, a team of scientists from the Physikalisch-Technische Bundesanstalt (PTB) (the national metrology institute of Germany) is conducting experiments with spheres of isotope-enriched silicon, which could be used as a new calibration standard. The experts, therefore, must determine the Avogadro constant, which indicates the number of atoms in one mole. "We calculate the number of atoms in a sphere and use mathematical methods to obtain the number of atoms per mole. In simple terms, we find out how much a silicon atom weighs and through inverse conclusion can thus determine how many silicon atoms are needed for a kilogram," explains Dr. Ingo Busch, physicist at the PTB in Braunschweig. "The mole is the mediator between the atomic mass scale and the kilogram."

During the production of these spheres at the PTB, a natural oxide layer of silicon dioxide (SiO_2) is formed. This also has an influence on the mass and volume of the silicon spheres. The problem is that the native layer grows slowly and, in part, unevenly. This makes it very difficult to measure the actual weight of both the oxide layer and the sphere. Therefore, an alternative, homogeneous coating is required to reduce measurement inaccuracies and precisely determine the volume and mass of the sphere.

Alternative SiO_2 layer minimizes measurement uncertainties

Researchers of the neighboring Fraunhofer Institute for Surface Engineering and Thin Films IST have succeeded in coating a silicon sphere with such an SiO_2 surface that it

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meets highest standards. "With our method, we can apply a layer of SiO_2 with a precisely defined roughness and an adjustable layer thickness to the sphere. In addition, the layer is also stoichiometric, which means that the ratio of the individual atoms remains constant among each other or the ratio between silicon and oxygen," says Tobias Graumann, a scientist at IST.

For the coating, the researchers at the IST selected the Atomic Layer Deposition (ALD) (see box "Atomic Layer Deposition ALD"). The advantage of this method: A reproducible, extremely thin oxide layer with a homogeneous thickness can be applied to the sphere. Potential impurities, such as carbon or nitrogen, are below the limit of detection. The roughness of the layers remains below a nanometer. "The sphere's roughness is not significantly increased by the coating. This is a factor which keeps the measurement inaccuracy below 10 micrograms. Even a fingerprint weighs more," says Graumann. And the time factor also plays an important role. By applying SiO_2 , the manufacturing process can be accelerated. In contrast, the growth of the native oxide layer can take several months.

Coating in clean room atmosphere

The ALD coating plant installed at the institute was specially adapted and prepared for the project so that all work on coating could take place in a clean room atmosphere. The main focus over the years of research was on how the silicon sphere was mounted in the reactor. Since the sphere has to be coated over the full area, the researchers decided to use a three-point mounting, such that the object to be measured makes contact at three points. "Here, we take advantage of the ALD's mechanism: the gaseous chemicals typically diffuse between the sphere and the three contact surfaces of the mount, which are also coated in the process," says Tobias Grauman.

The coatings of the silicon sphere have been concluded and now the measurements are being performed at the PTB. The results will be available this summer and will then be presented at the Conference on Weights and Measures in autumn 2018. Then, at the latest, the new sphere will replace the original kilogram as the standard. At the metrological meeting a decision will be made on the redefinition of the kilo.

The researchers at the Fraunhofer IST and their colleagues at the PTB hope that the silicon spheres will become the new calibration standard. Metrology institutes and calibration laboratories will be given the opportunity to acquire copies of the spheres. PTB plans to offer three different price and quality variants.

The SiO_2 developed at the Fraunhofer IST can be applied not only to spherical systems, but also to any complexly structured surfaces. The fields of application are diverse and range from optical applications through the semiconductor and electronics sectors to photovoltaics.

Atomic Layer Deposition (ALD)

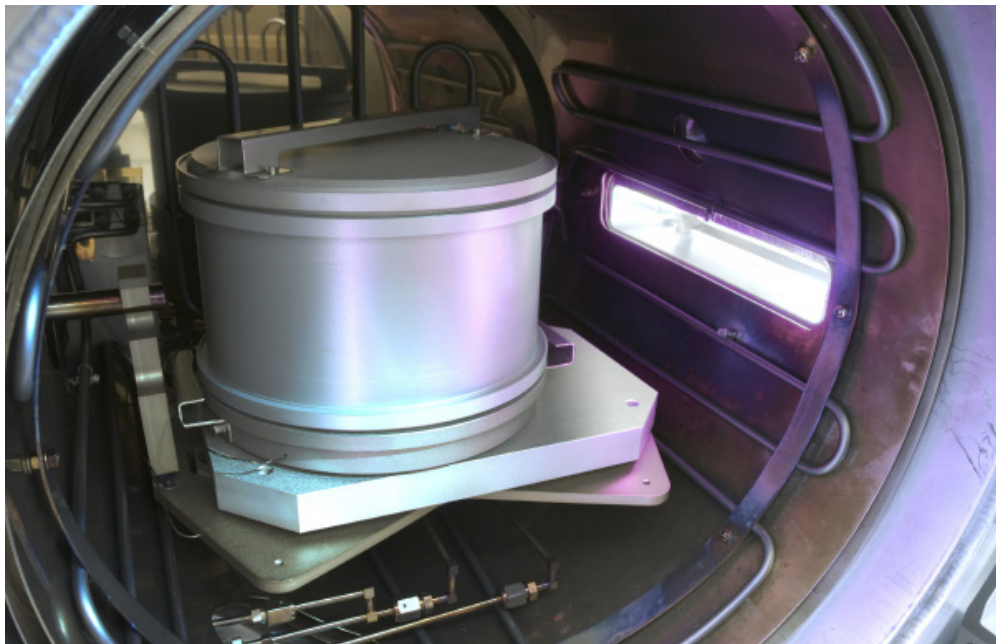
If individual atomic layers are to be applied with extreme precision, then the Atomic Layer Deposition (ALD) is the method of choice. Layer by layer, the coating is gradually applied – the layers are virtually stacked onto the surface. This is a protracted process requiring numerous coating cycles.

BCoating process at Fraunhofer IST

The layers are formed by a chemical reaction based on two substances, which are successively admitted into the reaction chamber. The Fraunhofer IST also follows this principle: to create the SiO₂ layer (see text), Tobias Graumann and his team apply the easiest method, which entails admitting two chemical substances in gaseous form into the coating chamber. This leads to two successive, self-limiting surface reactions. In the first step, the surface in the chamber is exposed to reactive molecules in the gaseous phase. When reacting with the surface, only an intermediate product is formed – the first monolayer. The researchers flush the excess molecules and resulting by-products out of the chamber using nitrogen – overdosing can thus be avoided and the reactions are separated from each other. The second chemical is allowed into the chamber and reacts with the intermediate product. The desired coating product remains on the surface. If this reaction occurs on the entire surface, further chemicals can no longer adhere to the chemically saturated surface. Experts therefore speak of a self-controlled, easily controlled growth – the main feature of the ALD. This reaction cycle can be repeated as often as necessary to achieve the desired coating thickness.

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Inside the ALD coating plant at the Fraunhofer IST: the coating chamber for three-dimensional objects © Fraunhofer IST | Image in color and print quality: www.fraunhofer.de/presse



The silicon sphere to be coated must be handled like a raw egg. On the right in the picture: one of the three-point mounts. © Fraunhofer IST | Image in color and print quality: www.fraunhofer.de/presse

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