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Innovative mass standards for the worldwide transfer of the redefined unit kilogram

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Abstract

Manufacturing processes and handling procedures for new stable silicon mass standards with unprecedented accuracy have been developed and transferred to industry. The patented manufacturing process and the high precision calibration procedures were developed by Physikalisch-Technische Bundesanstalt (PTB). Both the manufacturing process for the spheres and the handling of the spheres were extended to two small and medium-sized enterprises (SME). The transfer was part of the three-year transfer project "Si-kg" for industrial requirements and placed on the market with great success.
Depending on the custom requirements, two different qualities are offered within a newly established sales infrastructure. One potentially primary quality derived from volume and density and another high quality which serves as high precision mass standard. This coincides completely with the revision of the International System of Units in 2019 and the resulting new possibilities to disseminate the unit Kilogram for a practice oriented and stable traceability chain.
As a result of the project, several silicon spheres were sold before the end of the project. Further inquiries reflect the increasing market for high precision mass standards.

Kilogram, Si, mass standard, dissemination

1. Introduction

The Physikalisch-Technische Bundesanstalt (PTB) was significantly involved in the redefinition of the International System of Units [1]. In the field of mass, for example, the 130-year-old International Kilogram Prototype (IPK) was replaced by kibble balance methods and by several unique spheres with nominal mass of 1 kg made of monocrystalline isotopically enriched silicon (^{28}Si) [2]. In case of the silicon sphere, the unit kilogram is linked to the mass of its individual atoms and "traced back" to them [3]. A key challenge of this new approach has been to produce spheres with unprecedented accuracy in roundness and surface quality. In order to minimize measurement errors and undesirable environmental conditions, many physical barriers had to be overcome. For this purpose, PTB developed a manufacturing process for monocrystalline silicon. Since the high-cost and the complex enrichment process of the isotopically enriched material, only 12 spheres made of ^{28}Si will be available in the world.

Therefore, alternatives had to be developed for National Metrological Institutes, Calibration Laboratories and manufacturers of weighing instruments to reliably disseminate the unit kilogram via silicon spheres.

2. Mass standards made of silicon spheres

Since May 20, 2019 the revised SI came into power. One essential realization in mass are spheres of silicon. Spheres of highly enriched isotopic monocrystalline silicon ^{28}Si of 1 kg nominal weight with 99.998 % enrichment [4] used for the realization of the mass derived on the value of the Planck constant h.

Silicon spheres made of natural silicon which are manufactured similar to the silicon spheres made of silicon 28 can be used as stable and high accurate mass standards.
All spheres can be marked in order to determine the location with is required for the volume measurement on spherical interferometer and oxide layer growth on XPS/XRF measurement device. The markings show the crystal orientations [100], [110] and [111] and are used as calibration points and for precise measurements. All markings are done with laser ablation and ensure minimum changes to the silicon surface.

3. Industrial manufacturing process for silicon spheres

The manufacturing process was transferred to a small and medium-sized enterprises (SME) in order to produce spheres of natural mon-ocrystalline silicon (^{nat}Si).

The patented manufacturing process of the purpose-built machine provides contaminant-free spheres showing only minor shape errors, low roughness and a very uniform and stable oxide-layer [4]. Despite the robust crystal structure, the polishing process enables due to the special composition of the polishing paste to physically remove atoms from the surface without scratches nor subsurface damage of the crystalline structure. The polishing process achieves very low roughness and achieves a defined thickness of oxide layers. The scratch-free surface prevents the diffusion of (foreign) atoms. As a result, mass standards made of silicon achieve a long-term stability that has not been achieved before.

The manufacturing partner is able to use the transferred process for the production of a large range of spheres. This includes spheres made of other materials as well as spheres that can be used as density standards.

4. Handling, transportation and cleaning of silicon spheres

The know-how about handling silicon spheres and the necessary tools were passed on to a second company which has a long experience in manufacturing weights. In a series of trials and practical tests lasting several years, PTB has identified materials with which silicon spheres can be safely handled, stored and transported. In order to guarantee the extremely high mass stability, the silicon spheres are mounted on special rings which neither scratch the sphere nor leave any adhering to the surface of the sphere.

In contrast to other mass standards, silicon spheres can be cleaned in a simple, efficient and cost-effective way to remove all surface contamination. This cleaning procedure enables the high stability. Therefore, special cloths and handling equipment were developed and optimized with the industrial partner. In addition to the spheres, the cleaning and handling equipment, the sales partner offers training courses for silicon spheres handling. In close cooperation a broad selection of accessories for silicon spheres like transport container and transport case, special tongs and fork and sphere holder for universal applications were designed [5].

5. Results

With the transfer of sphere manufacturing to an industrial company and the transfer of handling to a second SME, a worldwide unique ready to use infrastructure has been realised which allows highly stable and accurate mass standards to be delivered.

During the project period, the requirements for the high-precision manufacturing were even more than exceeded. Silicon spheres with form deviations of less than 20 nm can now be manufactured for the first time by industry. If the diameters of the sphere are plotted according to its spherical coordinates, the topography specific to this sphere is obtained. Figure 1 shows the data in Mollweide projection with a peak to valley value for the radius of 10.2 nm with U(k=2)= 2.3 nm.

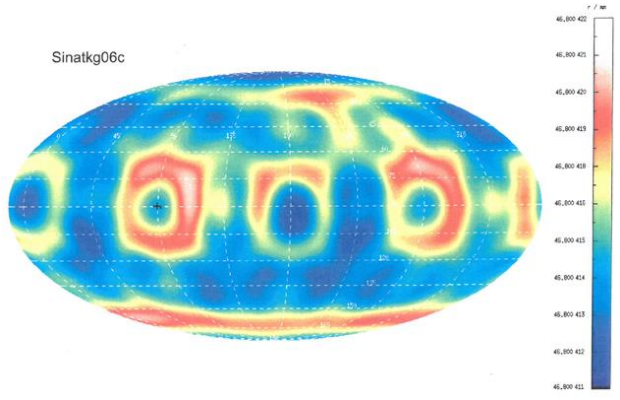

Figure 1. Topography: Mollweide projection of a silicon sphere manufactured of industrial partner

Table 1 shows the achieved measurement uncertainties of the silicon spheres manufactured by the industrial cooperation partner [6]. All spheres are adjusted to 1 kg +/- 200 mg. Table 2 shows the corresponding environmental conditions for calibration.

The established infrastructure can meet the increasing global demand for stable high-precision mass standards. This is underlined by the sales of several silicon spheres, which were already sold to international customers during the project period. In addition, more than 12 spheres were distributed to national metrology institutes worldwide for individual practice.

Table 1 Measurement uncertainties U(k=2) of silicon spheres manufactured by industrial partner. All measurements are done by PTB

Parameter	U(k=2)
Mass in air	50 µg
Mass in vacuum	25 µg
Density (hydrostatic)	0.004 kg/m³
Roughness S_a	< 0.5 nm
Form deviation RONt	10.2 nm
Average layer thickness	0.4 nm

Table 2 Environmental conditions and specifications for calibration of silicon spheres at the highest level

Measurand	Specification
temperature U(k=2) = 0.02 °C	19 °C – 22 °C
temperature drift U(k=2) = 0.02 °C	±0.05 °C/h
temperature stability U(k=2)	±0.1 °C/24h
humidity	45% - 55%
humidity drift	±1%
air pressure	0.06 mbar
mass comparator electrical weighing range resolution linearity standard deviation	 1.5 g 0.1 g ≤ 2 µg 0.4 µg at 1 kg

6. Summary

Processes for the production of mass standards made of highly enriched silicon with nominal mass of 1 kg have been developed in the preparation of the redefinition of the unit Kilogram. The methods and procedures were successfully transferred to industry. The transfer to two SMEs of the spheres led to previously unattained small form deviation of less than 20 nm. An infrastructure which includes user-friendly handling tools and cleaning procedures was established. In future the procedures can be modified in order to manufacture spheres made of different materials and with different diameters.

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