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Improved manufacturing process chain for silicon spheres

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Abstract

With the successful reworking of two silicon ^{28}Si spheres for the International Avogadro Coordination (IAC) project at the Physikalisch-Technische Bundesanstalt Braunschweig (PTB), the manufacturing chain was improved, furthermore, with respect to the achievable form deviation.
All spheres manufactured and reworked at PTB show sufficient surface quality, e.g. average roughness values of approx. 0.2 nm and below. The surfaces are free from defects, metallic contamination and subsurface damage. To reduce the measurement uncertainty of the subsequent volume determination, which is a crucial factor of the Avogadro experiment, a further decrease of the form deviation of the sphere was mandatory. Results of the refined process chain are presented. A smallest form deviation of 16.3 nm could be achieved so far.

Superpolishing, Silicon, Subsurface Damage, Manufacturing Chain, Avogadro Project

1 Introduction

The International Avogadro Coordination (IAC) targets the redefinition of the unit kg by means of natural constants to replace an artifact, made of PtIr more than 100 years ago, to define units, for details, see, e.g. [1]. However the technical tool for the metrological realization is a sphere made of silicon, which in the ideal case has a perfect lattice, has no form deviation or surface irregularities nor metal contaminations in the surface and in the end, has stable and well-defined surface layers. From the manufacturing standpoint, this task turned out to be very challenging. E.g. unacceptable metallic contamination was detected in the surfaces of ^{28}Si spheres, as they were manufactured in Australia.

It was then decided to rework the spheres at PTB. In a first step, metallic contamination was removed by means of an adequate etching procedure, but the surface topography and the form deteriorated. In parallel, a new manufacturing chain was developed to improve surface integrity and form. The herein presented results are part of the reworking process of the ^{28}Si spheres "Avo28 S5" and "Avo28 S8" [1].

Due to the excellent infrastructure of the PTB concerning measuring expertise, a valuable feedback is available for developing and establishing new manufacturing technologies. Measuring and manufacturing are strongly interrelated, as the manufactured artifacts are to be measured. Close collaboration is essential to bring the intention of the metrology and the capabilities of manufacturing to congruence. The know-how, obtained in the long-term work at PTB is very important for developing the manufacturing chain proposed here. Several optimisations are still under development and a further reduction of the form error is expected, finally limited by the characteristic change of properties of an Si crystal subject to its orientation.

2 Manufacturing chain

Mono-crystalline silicon has several drawbacks for manufacturing, e.g. high mechanical sensitivity of the brittle and highly pure material, which easily leads to cracks through the lattice and the whole workpiece. Furthermore, the anisotropy of the crystal is present in all existent orientations on a sphere. Material behavior, e.g. Young's modulus and hardness, and thus the removal rate is position dependent. Finally, structural changes of silicon may occur due to high local pressures and temperatures on surface, e.g. amorphous phases, various residual crystalline structures or dislocations.

Several steps of processing, which are common in conventional optics and precision manufacturing [2], are carefully adopted for the manufacturing chain proposed here. For a general map of single step results, see figure 1. A main classification of the manufacturing steps can be outlined by means of the tools in contact with the workpiece.

Figure 1. Hollowed ingot, cut, turned, lapped and polished sphere

While performing coarse preparatory work, the processes use fixed and bonded tools. In the subsequent step, loose grain of decreasing size is used for lapping operations with rigid tools and finally polishing operations are applied to improve form and surface continuously.

2.1 Coarse Machining

In the first manufacturing step, a cylinder is cut out of the ingot by means of a diamond plated hollow drill. The diameter of the ingot must be large enough to facilitate a reserve of several millimetres of the twice cylindrically cut workpiece for further processing. In the second step, the rough cut form is turned into a spherical shape in a conventional lathe by means of a polycrystalline diamond cutting tool. It is turned in two succeeding steps, cemented into an auxiliary tool to be fixed on the chuck. The form deviation is then typically less than 1 mm.

2.2 Lapping Processes

The following lapping processes incorporate several grain sizes of alumina. For coarse grains the laps are made of metal and for smaller grains (below 5 µm in size) they are made of glass. All media are used in an aqueous solution. Some information about the earlier manufacturing of the Avogadro spheres was published by the Australian project partners, e.g. see [3]. Lapping operations are partially similar due to comparable tools and materials.

The matte appearance of a coarsely lapped sphere is shown in the centre of figure 1. The mechanism of lapping is a combination of the mechanical impact of the temporary fixed and the rolling grain onto the workpiece. High mechanical stresses induce damage in the bulk material.

Breaking loose, rolling, scratching and even ploughing are the mechanisms active for a high removal rate. With smaller grain sizes the rolling movement of the grain is predominant.

Each grain size has to remove the damage in the surface of the previous step completely. A rough rule for required removal could be to remove at least five times the diameter of the average grain size of the previous process step. This leads to a removal of the subsurface damage of the previous process. After fine lapping, the form error typically is in the order of 100 nm. It is determined by means of a form measurement device. A reserve of several micrometers is provided for the subsequent polishing operations.

2.3 Polishing Processes

For the non-deterministic machining of the ultrasmooth surfaces, polishing pitch was used. A medium hardness blend of commercially available optical polishing pitches was established for the polishing operations [4, 5].

In the first polishing step, colloidal alumina of a mean grain size of approx. 1 µm is used, and for the finishing process titanium dioxide with a mean particle size of approx. 100 nm in aqueous suspension is used. The result is a surface with a low surface roughness average down to 0.2 nm and below [5]. Processing times of several weeks with extremely low removal rates of just several nm/min indicate a novel physical removal mechanism. The superpolishing process [5] here proposed may work similarly to the assumed modes of action in “Elastic Emission Machining” or “Float Polishing” in [1].

3. Results

The diameter topography plots of both reworked ^{28}Si spheres “Avo28 S5c” and “Avo28 S8c”, and an additional new sphere of natural silicon “Si 12-05” are shown in the Mollweide plots of figures 2 to 4. For details of the interferometric volume determination at PTB working group 5.41, see, e.g. [6]. Please note that the same gray scales in the figures represent different spans of diameter variations in the plots, as the PV values indicate. The crystal lattice predominantly affects the amplitude and regularity of the form deviation due to the characteristic dependence on orientation. The larger the form deviation, the clearer the visible influence of the lattice, see figure 2. The clear rhombic dodecahedron of figure 2 seems to become less defined, the lower the form deviation is. The dodecahedron is typical for the cubic crystal class of the silicon.

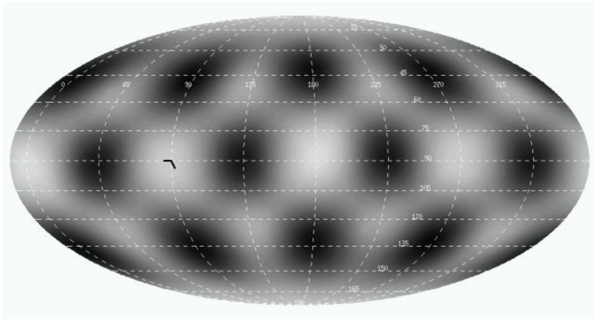

Figure 2. Diameter Topography “Avo28 S5c” , PV **34.6 nm**

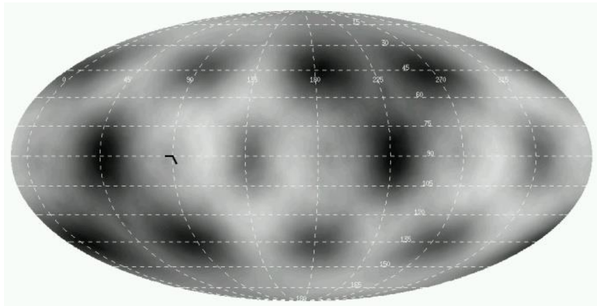

Figure 3. Diameter Topography “Avo28 S8c” PV **18.9 nm**

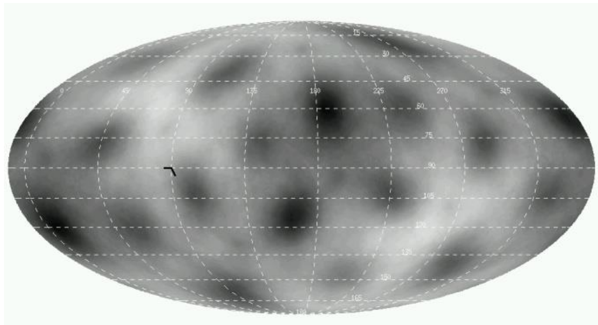

Figure 4. Diameter Topography “Si 12-05” PV **15 nm**

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References

[1] Becker P, Friedrich H, Fujii K, Giardini W, Mana G., Picard A, Poh H J, Riemann H, Valkiers S 2009 *Meas. Sci. Technol.* **20** 092002 (20 pp),

[2] Evans C , Paul E, Dornfeld D, Lucca D A, Byrne G, Tricard M, Klocke F, Dambon O, Mullany B A 2003 *Annals of the CIRP* **52**(2) 611–633

[3] Leistner A J, Giardini W J 1994 *Metrologia* **31** 231-243

[4] Meeß R, Feist C, Löffler F 2013 *Proc. EUSPEN 13th Int. Conf.* Vol. II, ed R Leach and P Shore (Bedford: euspen) pp 185-188

[5] Meeß R, Felgner A, Hinzmann G, Lück A 2014 *Proc. EUSPEN 14th Int. Conf.* Vol. II, ed R Leach (Bedford: euspen) pp 99-102

[6] Bartl G, Krystek M, Nicolaus A, Giardini W J 2010 *Meas. Sci. Technol.* **21** 115101 (8 pp)