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CO_2 snow cleaning of miniaturized parts

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ABSTRACT

The surface cleaning of components with sub-millimeter features poses a challenge in many applications. Challenges may be strong particle adhesion at the microscale, the difficult handling of the samples, the small size of the adhering particles, and unfavorable geometry. However, components often need cleaning before processing, assembly, packaging, applications, and measurement can proceed. Previous evaluations of CO_2 snow cleaning almost exclusively refer to the cleaning of smooth, flat or convex surfaces; typically examined samples were wafers and optical components. In this paper, we studied the CO_2 snow cleaning of complex surface shapes as found on microgears. The performance of CO_2 snow cleaning is compared with ultrasonic cleaning and a high-speed air jet. The evaluation of the 72 cleaning experiments is based on the optical microscope images of the four investigated samples before and after cleaning. Results show that CO_2 snow cleaning removed more than 95 % of the micrometer-sized contamination from our test samples. Ultrasonic cleaning removed 88 % of the particles and the high-speed air jet removed 74 % of the particles.

1. Introduction

Metrology strives for reliable measurements of quantities with small uncertainties. Small measurement uncertainties and consistent measurement routines, for example, help industries to reduce costs by optimizing quality control. Particles on measured samples or relevant parts of a measurement machine such as tactile probes may distort a measurement result. This is particularly true of precision measurements at the microscale [1].

Contamination may impair a probing system because adhered particles cannot be removed without destroying the probe or because the particles have already damaged the probe [2,3]. Precision cleaning is an economic issue as well since microprobes used for high-precision measurements are costly and may become unusable when seriously contaminated. The time needed for cleaning often exceeds the time needed for other preparations like setting up software. Most measurement standards are treated with oil (for example, petrolatum or silicone oil) or anti-corrosive wax for conservation. These standards need routine cleaning before every measurement. In practical metrology, cleaning the sample prior to the measurement is a vital step.

In micrometrology, the desired small measurement uncertainties lead to high requirements on sample and probe cleanliness, since the particles negatively influencing the measurement are typically of similar dimensions to the desired uncertainty and larger. Fig. 1 illustrates the impact of contaminated samples on tactile measurements. Similar problems are well-known in metrology – for example in atomic force microscopy.

Geometries like holes and concave structures, and the need for rotating the sample to allow cleaning from all directions make cleaning three-dimensional microstructures more difficult than cleaning wafers or other flat surfaces. The goal of our work was to test and compare the cleaning efficiency of CO_2 snow cleaning during controlled experiments and to analyze the parameters that influence cleaning efficiency.

CO_2 snow cleaning is a fast, dry, cryogenic, aerosol cleaning process. Fed by pressurized CO_2, a nozzle ejects a mixture of solid CO_2 *snow* and gaseous CO_2. This high-velocity gas/particle stream strikes contaminated surfaces and removes contaminants and adhering particles. The cleaning mechanism relies on mechanical, thermal, and chemical effects. The CO_2 snow transfers momentum to overcome particle adhesion; the CO_2 snow at a temperature of $-78.5\,^{\circ}C$ freeze-fractures non-hydrocarbon based organics; the moving CO_2 gas carries away liberated particles; the solvent action of liquid CO_2 removes hydrocarbon films [4]. CO_2 snow particles liquefy temporarily when they hit the surface and are pressurized. The cleaning process is residue-free since CO_2 becomes gaseous at room temperature and atmospheric pressure. Several papers [5–8] report on over 99 % sub-micrometer particle removal.

Several factors influence the cleaning efficiency. Yang et al. [9] report on optimized cleaning efficiencies for a 15° incident angle, a 40-millimeter cleaning distance, a 0.3-millimeter orifice size, and a 50-

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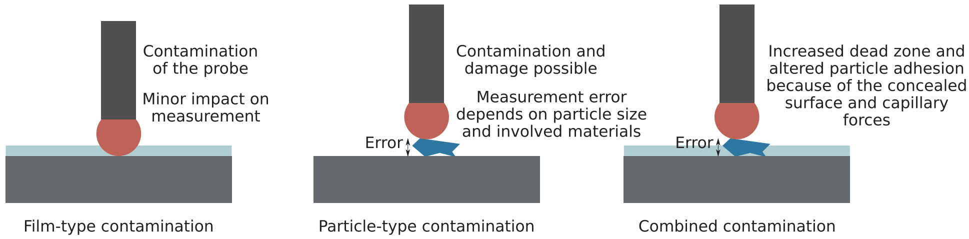

Fig. 1. Types of contamination and their effects on measurement and cleanability.

millisecond time base. However, these values depend on the equipment used and especially the nozzle design, which is not documented by the authors in detail. Heating the sample (about 50 °C), introducing nitrogen, and cleaning in a dry box are common techniques to avoid condensation and dry ice build-up [10,11,7]. Low humidity levels (20 % – 40 %) reduce the adhesion of particles [12]. Though, this leads to increased electrostatic charging. The CO_2 should be purified [10,13,6] and ultra-clean applications demand electropolished stainless steel tubing and valves to avoid contamination of the CO_2 [4]. Sherman [14] provides a comprehensive overview on CO_2 snow cleaning. Several theoretical [15–17] and experimental [18–20] studies of CO_2 snow generation and on cleaning mechanisms [21–23] have been published. Applications range from optics [24,25], electronics [26–28], metrology [8], contamination control [4], medicine [29], biotechnology [30], food refrigeration [31], pharmaceutical granulation [32], and art [33] to photovoltaics [34]. Researchers recently proved the good applicability of CO_2 snow cleaning on tactile probes as used in dimensional metrology [35–38]. For precision cleaning of microcomponents, the cleaning process must preserve fragile structures. Modifying the samples is unacceptable in metrology. CO_2 snow cleaning is damage-free for different materials (including glass and wafers) and most applications [14,10,8].

2. Methods and experimental setup

This study quantitatively compares three different cleaning methods, namely CO_2 snow cleaning, ultrasonic cleaning, and high-speed air jet, under repeatable conditions. The following samples were studied regarding their cleanability:

- Gear1: external spur microgear (metal, module 0.4 mm, 17 teeth)
- Gear2: internal spur microgear (metal, module 0.08 mm, 254 teeth)
- Nut: screw nut (brass, M4)
- Bore: bore (Y-TZP ceramic, Ø 3 mm, height 6 mm)

The samples vary in material, geometry, and accessibility of the cleaned features (see Fig. 2). Gears are highly suitable samples for this study since they are well-known industrial parts, their use in drive technology requires high-precision measurements and, thus, precision cleaning, and they possess a difficult-to-clean geometry in comparison to wafers and optical components which have mostly been studied for

Fig. 2. Overview of the cleaned samples.

CO_2 snow cleaning in the past.

Four types of particle contamination were analyzed:

- **Al-gran**: aluminum granules
- **Al-400**: alumina (Al_2O_3 with mesh size 400, volume weighted mean diameter 24.5 µm)
- **TiO2**: titanium dioxide (volume weighted mean diameter 28.5 µm)
- **SiC**: silicon carbide (volume weighted mean diameter 58 µm).

We chose these types of contamination because they cover a variety of sizes, materials, and shapes (see Fig. 3).

The contamination for the examined cleaning processes contained the following steps. First, the particles (~5 g) and an adhesive oil type CGLP (~15 g *Deganit BW 220*) were mixed. Second, the sample was immersed and the container was shaken for about thirty seconds. Third, the sample was removed and placed on a blanket to drain for five minutes. This procedure yielded a repeatable and uniform contamination with a lot of particles. Nevertheless, most particles have only loosely adhered to the sample surface. We used fully focused images from a digital, optical microscope (*Keyence VHX-5000*) to ensure the success of the contamination process and as a basis to calculate the removal efficiencies. All equipment that we used for the experiments is commercially available. Fig. 4 summarizes the procedure of the experiments.

Overall, the study comprised 72 experiments. We conducted 48 experiments to compare CO_2 snow cleaning with ultrasonic cleaning and a high-speed air jet. An additional 24 experiments performed with *Gear1* and *Gear2* focused on how the sample geometry and the particle size influence the CO_2 snow cleaning efficiency.

For every experiment, we took one microscope image (x200 magnification) before and after cleaning of the same area on each sample: The chosen designated areas were easy to retrieve and to reach with the microscope (see Appendix in Supplementary material for examples). For the analysis comparing the three cleaning methods, particles were counted by hand. The additional experiments on *Gear2* focusing on the particle size distribution were analyzed automatically by the microscope since the software allows for particle extraction and acquisition of other data like diameter and area. Unfortunately, this automated routine was only possible for the combination of *Gear2* and the alumina particles since in this case the edge detection succeeded.

2.1. *CO_2 snow cleaning experiments*

We used the *K1 standard unit* from *Applied Surface Technologies* with an additional ball valve for fine adjustments of our continuous CO_2 snow stream (see Appendix in Supplementary material) fed with high-purity, gaseous CO_2 at 57 bar. We used CO_2 grade 4.5, although a cheaper grade may suffice for this application. The nozzle has a 0.4 mm internal orifice diameter in the Venturi tube followed by a sharp nose cone. Cleaning of a sample took about thirty seconds.

The CO_2 snow cleaning results predominantly depend on the CO_2 snow particle size and speed because momentum transfer is the main cleaning mechanism for particles. Thus, we measured CO_2 snow jet velocities to further characterize our cleaning process. A high-speed camera (*Photron Fastcam SA-Z*) recorded 100 000 frames per second of a