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CONTACT MATERIALS FOR MASS ARTIFACTS

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ABSTRACT

The present work deals with the comparison of the properties of different contact materials in combination with a stainless steel mass artifact. Furthermore measurements for typical contact materials in combination with silicon surfaces with different configurations of the surface oxide layers are presented. An experimental setup for reproducible measurements of short to mid-term wear is introduced. The device allows the simulation of 10000 load cycles in less than six hours, while ensuring well defined contact motion. From the theoretical investigation of the contact physics and the measurement results recommendations for the design of contact points will be derived.

1. INTRODUCTION

As part of the ongoing efforts to redefine the SI based units by fixing natural constants [1] the unit of mass, which is currently the only unit still based on an artifact, will be defined by a fixed value of the Planck constant. There are two experiments for determining a value for the Planck constant, the Avogadro-Project [2] and so called Watt-balances [3]. Since both experiments are carried out in vacuum, after the redefinition the new kilogram will also be realized in vacuum. Thus, for connecting the new definition to the current definition, which is the International Prototype Kilogram (IPK) stored in air, air-vacuum transfers of mass artifacts are necessary. Also for disseminating the mass scale, at some point of the dissemination chain transfers from vacuum to air will be inevitable. In this context the continuity of the mass scale is a major concern [4]. The metrological behavior of the mass artifacts in such air-vacuum or air-inert gas-vacuum transfers is significantly influenced by their surface, as it will be covered by adsorption layers of water and hydrocarbons [5]. To improve the mass stability one approach is to use high quality surface finished bulk materials of high density. Another approach is to apply surface coatings to realize high quality mass standards. The use of bulk materials with high density and good magnetic properties in combination with inert and highly mechanically stable surface finishes allows producing highly optimized artifacts at reduced cost.

For the mid and long term mass stability of the artifacts it is desirable to reduce the wear during transport, handling and use in mass comparators. Aside the influence of abrasive and additive wear on the mass, the change of the surface parameters can lead to mass variations. The effect of changes of the surface roughness is expected to have a small effect on the adsorption behavior [6, 7]. The influence of damages to the stable oxide layer on silicon mass artefacts and especially to coatings is relatively unknown since damage in a microscopic area can change the chemical properties of the artifact dramatically.

The wear depends on the artifact material to contact material combination as well as the geometry of the contacting surfaces and the contact motion (maximum force, directional components).

In the following section the mechanical contact of weights during typical use will be analyzed and possible material combinations will be listed. In the third part of the paper a load changing setup for the experimental simulation of wear is described. The results of a first set of measurements is then discussed and finally conclusions concerning the design and material combination for contact points are drawn

2. MECHANICAL CONTACT OF WEIGHTS

2.1 Theory

During the use of weights for mass comparisons or calibration tasks, handling and transport of the mass artifacts is inevitable. This implies mechanical contact to the surface of the weight, and also other effects due to changes in surrounding conditions. Effects of surrounding conditions on water and carbonaceous surface layers have been extensively investigated, as well as contaminations on surfaces of weights. The question rises in how far mechanical interaction alters the surface and thus the metrological properties of the weight.

In case of adhesive wear, a contamination of the weight by material from the contacting surface occurs while in case of abrasive wear the surface of the weight is altered. The result can be a gain respectively a loss of mass and also a change of the sorption behavior in the contact region, also areas with increased surface roughness can tend to accumulate contamination.

The precise effects of mechanical interaction depend on the material combination (contact material / material of the weight), the geometries of the contacting surfaces, the maximum contact force and the direction of motion during contact. These factors are different for various mechanical interactions.

In transport containers weights are typically padded or fixed with softer materials with large contact areas. The forces during transport are undefined and the surfaces are constantly in contact.

In mass comparators the contact areas are typically small for different reasons such as to allow stable placing of the weights. During manual mass comparisons the weight is placed on the weighing pan by hand. The motion is undefined and not repeatable and the maximum contact force can exceed the full weight of the mass artifact. Also large lateral motion can occur. While for manual mass comparisons the number of load cycles is fairly limited, automatic load changers allow a large amount of load cycles. Also the motion is very well defined and thus the load changing is very reproducible for these systems. Depending on the construction of the load changer there is basically no, or very small lateral motion and by according control the contact force can be kept very low.

This paper will primarily focus on the mechanical interaction of weights in automatic load changers since these are commonly used in mass metrology at the level of precision where the addressed effects are of interest.

In precision mass comparators the weights which can be cylinders with flat bottom, knob weights with a concave bottom or silicon spheres are typically contacted by three spherical pins or three small cylinders. The contact of a small spherical pin to a flat surface can be calculated as Hertzian stress

$$p_{Hertzian} = \frac{1}{\pi} \cdot \sqrt[3]{\frac{1{,}5\,F}{r^2}\,\left(\frac{E}{(1-\nu^2)}\right)^2}\,. \qquad (1)$$

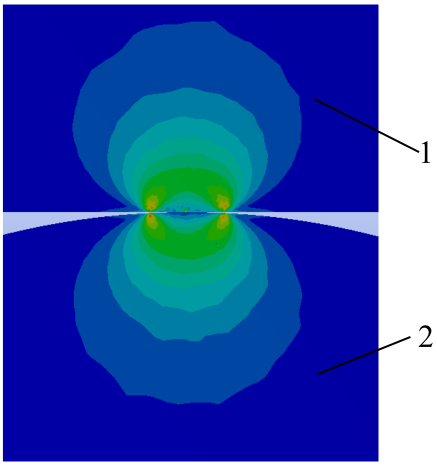


Figure 1: Distribution of stress for cylindrical weight (1) on spherical pin (2)

which considers the deformation in the contact region. (F – contact force, E – combined Young's modulus of pin and weight, ν – Poisson's ratio)

The distribution of stress, during mechanical contact of a weight with flat bottom on a spherical pin is shown in shown in Figure 1. If the stress exceeds the maximum permissible stress of either contact material or weight, even sub micrometer lateral motion can lead to deformation of the surface and also to abrasive wear (in case of the contact surface this can lead to additive wear to the weight). Hence a material combination has to be chosen so neither surface is damaged by placing the weight.

2.2 Material combinations

For mass artifacts the materials are chosen for mechanical and chemical stability of the weight surface, low electromagnetic (low suszeptibility and magnetization) and electrostatic (high conductivity) interaction. For mas comparisons in an air vacuum regime a high density is desirable to reduce the surface area and therefore the magnitude of adsorption effects. The international prototype kilogram and national prototypes are made from Pt/Ir, most commonly used weights are made of austenitic steel alloys. Other bulk materials are silicon, molybdenum and tungsten. An alternative is the combination of a bulk material with optimal density and electric properties with a surface coating for inert behavior and mechanical robustness. Such a combination is for example copper with mechanically polished rhodium coating.

In Table 1 a list of contact materials is given. For the use in balances the contact materials need to be conducting to avoid the buildup of electrostatic charges, while in storage vessels this is not an issue. These materials are widely used; one approach is to use soft plastic materials to avoid abrasion of the weight, while in some applications the contact points need to be highly stable themselves and are thus made of metal that is generally chosen to be softer than the weight.

*Table 1List of contact materials (non-conducting materials are marked by **)*

Material	**Radius of spherical pin**	**Providing Institution**
PEEK (mixed with carbon)	R = 2 mm	NPL
PTFE**		
AL		
Torlon**		
Ti	R = 4,2 mm	MIKES
PTFE**	R = 3,5 mm	
PEEK**		
TecaPEEK (with CNT)	R = 2,25 mm	TU-Ilmenau
CuSn8		

In Figure 2 the Hertzian stress for a one kilogram steel weight resting on three spherical pins, versus the Young's modulus of the pins is shown. The blue curve corresponds to a radius of the pin of 2.75 mm, for higher radii the stress decreases. For different materials the points denote the permissible stress at the x-coordinate of the material's Young's modulus. For acceptable values that ensure that neither the weight nor the pin are damaged the stress has to be below the permissible stresses of the weight as well as the pin. This is only the case for PEEK and PTFE at high radii.

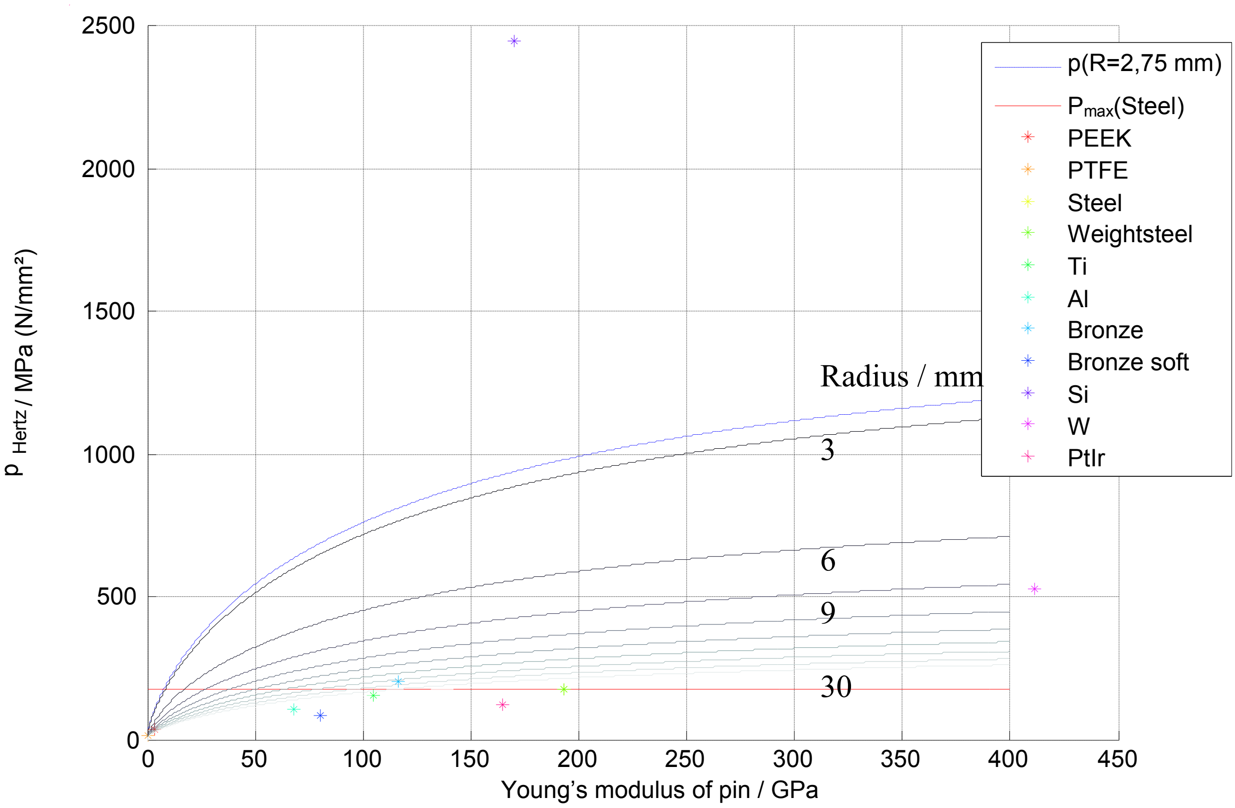


Figure 2: Resulting and permissible stress for stainless steel and various contact materials

The number of possible material combinations for the given materials for weights and contact points is quiet big for an experimental investigation, thus only two weight materials were used for first tests: stainless steel, as the most common and silicon, as the only non-ductile and non-metallic material.

3. MEASUREMENT SETUP

3.1 Apparatus

The aim of the experiments is to simulate the wear occurring on a weight, used in an automatic mass comparator within a medium timeframe of five to ten years. This corresponds to 1000 to 10000 load cycles, and a straight forward way is to continuously cycle the weight under test in the automatic load changer of a comparator balance with different materials used as contact points. However this approach has some disadvantages. The time for each load cycle cannot be reduced below a certain limit. Furthermore the relative motion of weight and contact points depends on the specific adjustment of the device, so that the obtained results are quiet instrument specific. For the present investigation a special setup optimized for wear measurements was designed (Figure 3). The contact points are realized as spherical pins, that can be easily manufactured from different materials and reduce the need of mechanical alignments. Also the area of contact is very well defined. The pins are attached to a parallel guide with flexure hinges. The guide is loaded with weights that define the contact force.

During contact the guide is not deflected, so that the spring constant of the flexure hinges does not contribute to the force. For load cycling the guide is deflected by a load changing mechanism consisting of a rotary dc motor and eccentric tappet. The dc motor allows exact positioning and very low contact velocities. A load cycle is completed within two seconds. The weight material samples are a cylindrical 100 g steel disc weight (diameter 40 mm) class E1 [8] clamped to an aluminum carrier and silicon wafers that are clued to polished steel carriers. The weight material samples are fixed to a linear stage for positioning relative to the contact pin. The position of the samples on the stage is given by dowel pins, so that the samples can be removed from the setup in between testing without affecting the contact area. Due to the fact that both weight- sample and contact pin are fixed in combination with the use of a monolithic parallel guide the contact motion is limited to the vertical direction with almost no lateral movement.

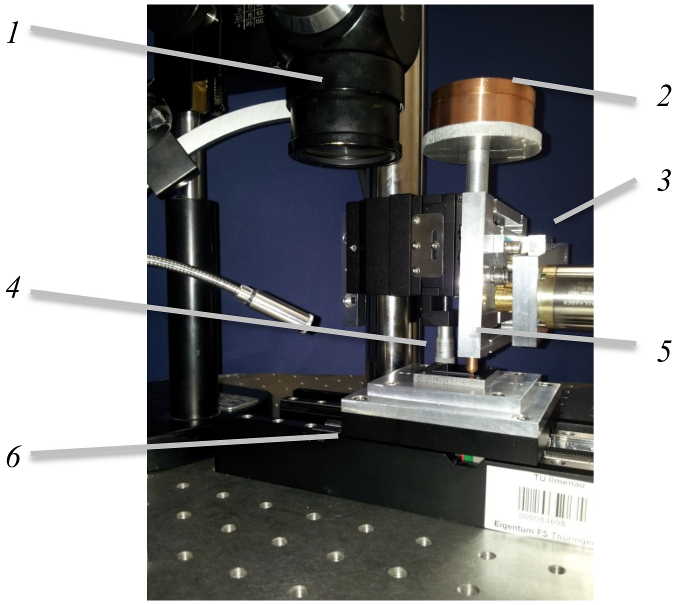


Figure 3: measurement setup: 1 – microscope, 2 – weight, 3 –parallel guide with load changing mechanism, 4 – Si surface sample, 5 –spherical pin (contact material), 6 – positioning stage

3.2 Experimental procedure

To establish the initial condition in a first step weight material sample and contact pin are cleaned with pure ethanol. To facilitate finding of the contact region the weight material sample is marked mechanically using a needle with a boron nitrite coated tip (radius 2 µm) and a micro manipulator. This is especially important in case of no visible contact marks but also in general the contact region and the corresponding marks are smaller than a diameter of 100 µm. After that the surface topology of both weight-sample and contact pin are measured. Contact pins made of soft materials such as PTFE and PEEK were visually inspected for contamination with hard particles from the manufacturing process. Then 1000 load cycles were performed in one contact region, with a load of 0.33 kg on the pin. Weight-sample and pin are analyzed again to evaluate the wear. To gain information on a possible contamination of the weight, for instance by debris from the pin, the weight-sample is cleaned and measured again.

The surface analysis was for most cases done in the NMM [9] using an autofocus probe [10]. For confirmation of the results the measurements on silicon were repeated using an atomic force microscope (AFM).

3.3 Data evaluation

For better comparison of the results the qualitative evaluation of the surface topologies needs to be complemented by numerical values. These have to be automatically generated to reduce the influence of manual evaluation and thus to improve the repeatability.

The numerical values chosen are the arithmetic mean surface roughness Ra and the surface roughness depth Rz. The evaluation of surface roughness [11] is not intended for the evaluation of localized surface perturbations, yet a modified evaluation gives reproducible results that correspond to the qualitative evaluation and allow a comparison of different material combinations.

For the evaluation in the scan the area of impact is selected and the derived matrix of x,y and z values with z being the profile height is processed. The digitization distance of the scans is 0.5 µm to 1 µm. The data is high-pass filtered (Gaussian) to receive the roughness profile from the primary data and very high frequency noise of the sensor is low-pass filtered (average) The roughness parameters are calculated from the matrix by treating every line as a single sampling length. So values for the x-direction are calculated, and in the same manner for the y-direction by regarding every column as a sampling length. The values for x and y direction differ for some materials such as steel due to an anisotropic surface structure (polishing marks), and are averaged to receive a single value to characterize the material combination. The calculated values for Ra are a measure of the integral change of the surface, while Rz emphasizes localized effects.

4. RESULTS

4.1 Different contact materials on Steel

In Figure 4 a typical measurement result is shown. After cleaning the impact mark is reduced, indicating that some contamination of the weight occurred.

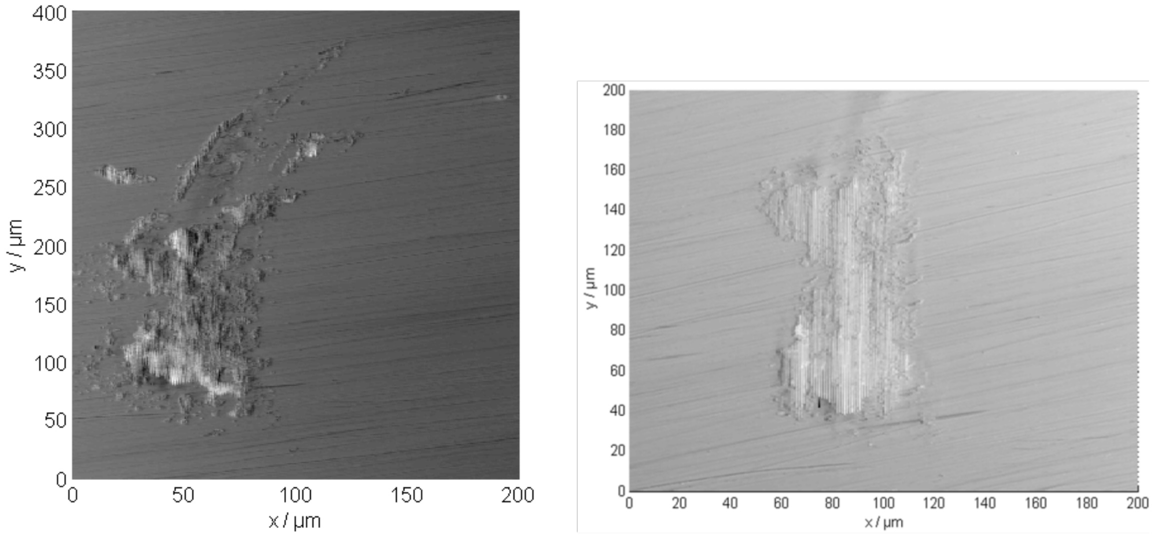


Figure 4: left- contact area of Aluminum on stainless steel after 1000 load cycle, right- surface after cleaning

An overview of the results is given in Table 2 and Table 3. For each contact material values are given before and after cleaning. The values for Ra_{max} and Rz_{max} are the limits for class E1 weights. Yet these values correspond to the condition of the entire surface and according to OIML R 111-1 small surface defects do not necessarily affect the classification of the weight. All materials show contamination on the weight. For PEEK and PTFE the contact region was not distinguishable from the unaffected material, so no additional value was given.

The overall influence of all materials besides Ti and Al is small but all metallic contact materials cause local, deep defects that lead to a large value for Rz. The same is true for TecaPEEK (PEEK TU-I) and Torlon. It is assumed that the reasons are the carbon nano tubes included in TecaPEEK and contamination of the Torlon.

Table 2: arithmetic mean surface roughness

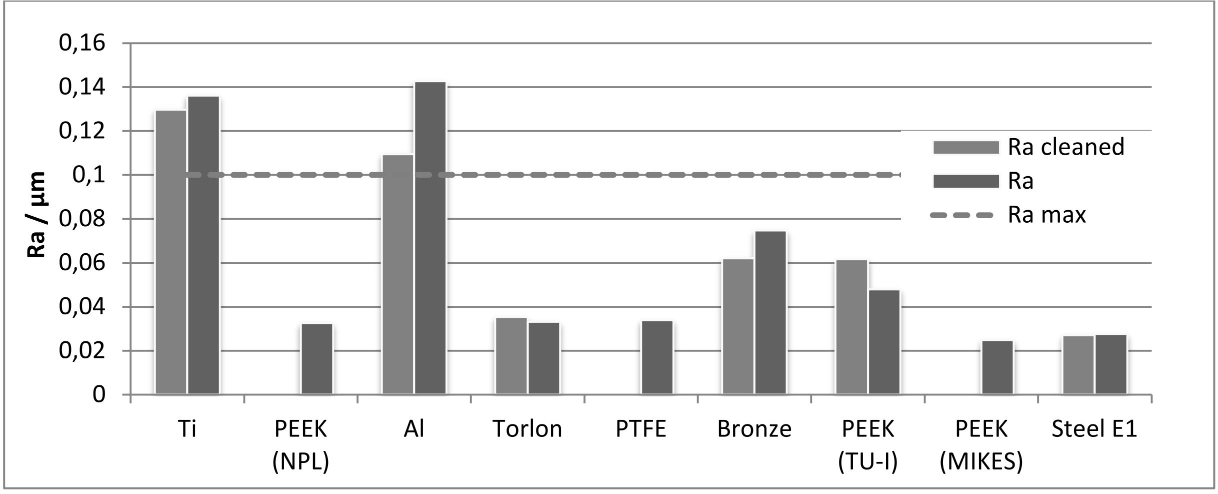
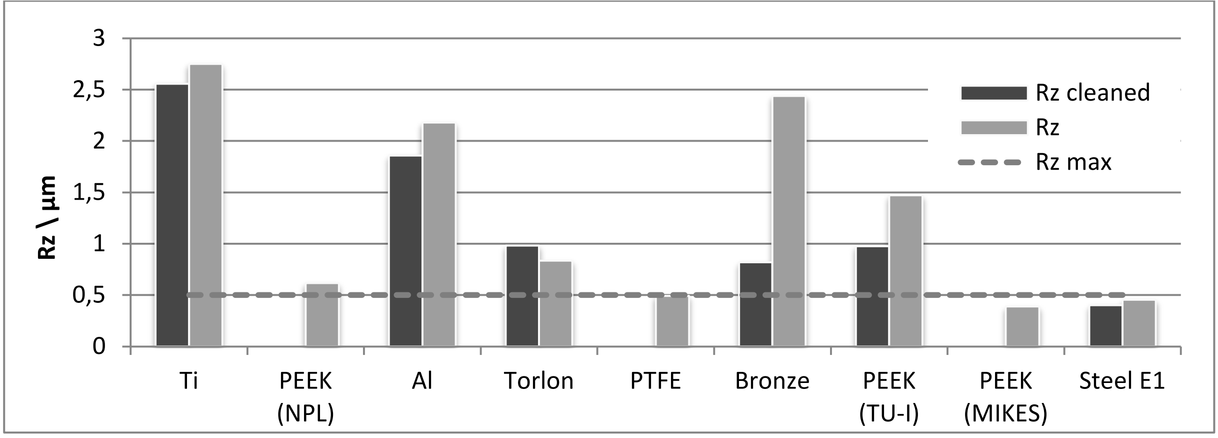


Table 3: surface roughness depth



The in section 2.1 predicted behavior for PEEK and PTFE was confirmed. Still the materials tend to contaminate the weight and are furthermore not suitable for the use in load pans of balances due being electrical insulators and low mechanical stiffness. On the other hand an application in transport containers is reasonable since this specific contamination can be removed and for transport containers the physical integrity of the weight and especially its surface are the primary aim.

The poorest results were achieved with titanium, the contact material with the highest stiffness. An issue to be addressed in this context is the surface quality of the contact pins. In general samples with high surface quality will behave close to the analytical model while poor surface qualities and related to that low load factors of the surface will lead to microscopic stress peaks. Depending on the material this will lead to increased additive or abrasive wear. In case of the titanium pin the surface roughness was Ra = 2 µm and Rz = 16 µm. The measurement for bronze was repeated after polishing the pin to Ra = 0.024 µm and Rz = 0.54 µm. As can be seen from the surface scans in Figure 5, even after 2000 load cycles no significant change of the weight surface occurred.

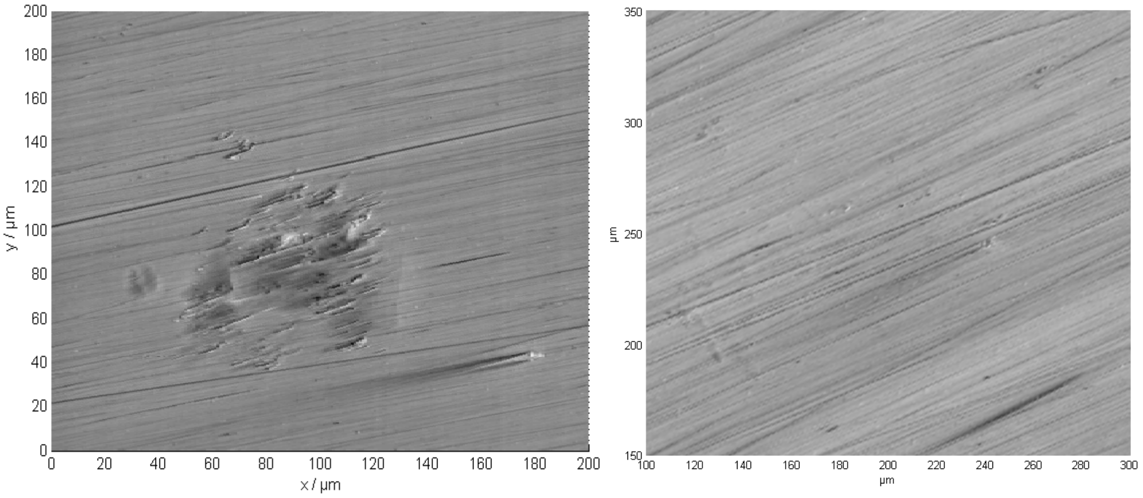


Figure 5: left- contact area of bronze on stainless steel after 1000 load cycles ,right- polished bronze after 2000 load cycles

4.2 Silicon

The measurements were repeated for Silicon as weight-sample in the same manner as described above. Instead of using a large number of contact materials only TecaPEEK and Bronze were used but various surface configurations of the silicon surface were tested. The weight samples include Si with 5 µm and 10 µm artificially grown oxide layer and natural oxide layer.

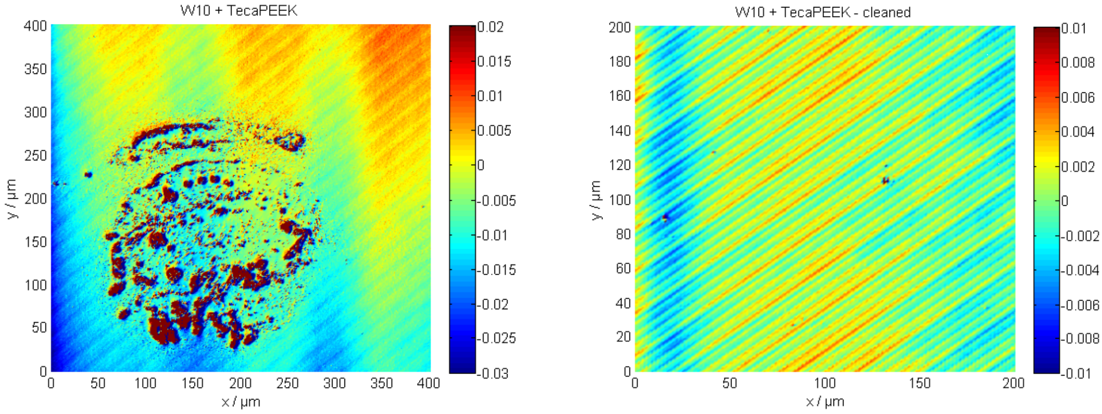


Figure 6: left – TecaPEEK on Si with 10µm oxide layer after 1000 load cycles, right – surface after cleaning

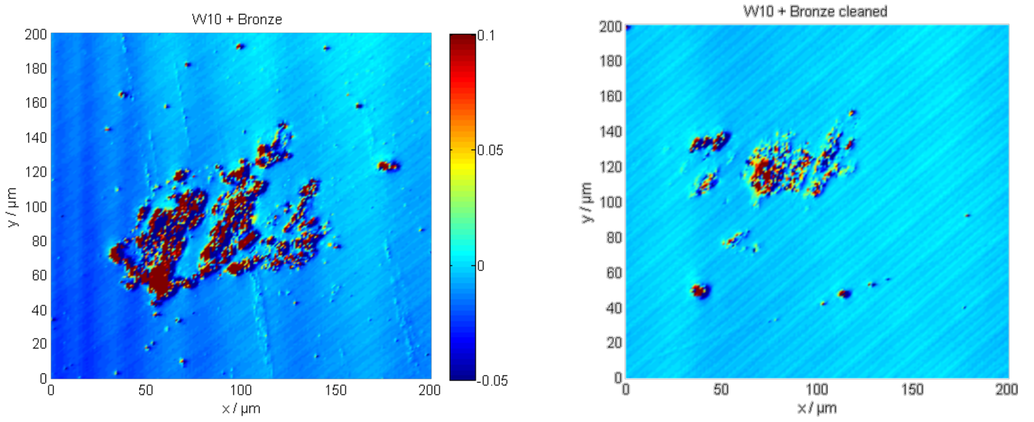


Figure 7: left – bronze on Si with 10µm oxide layer after 1000 load cycles, right – surface after cleaning

In figures 6 and 7 exemplary results are shown. Again as on steel the surface is contaminated after load cycling. The traces of TecaPEEK could be completely removed while bronze caused some surface defects. On the cleaned samples the scans show the surface structure of the silicon, caused by the manufacturing process.

Table 4: arithmetic mean surface roughness

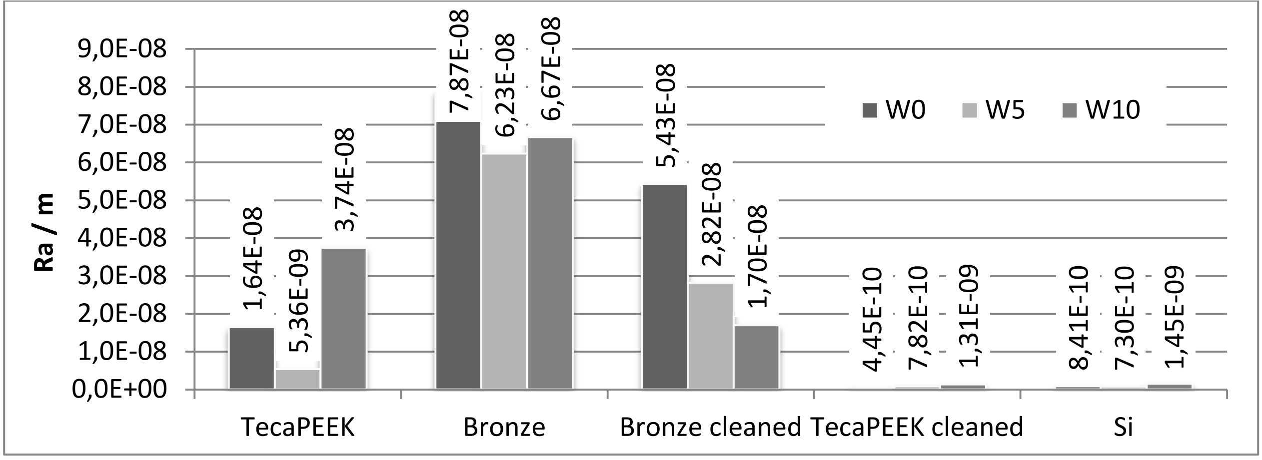
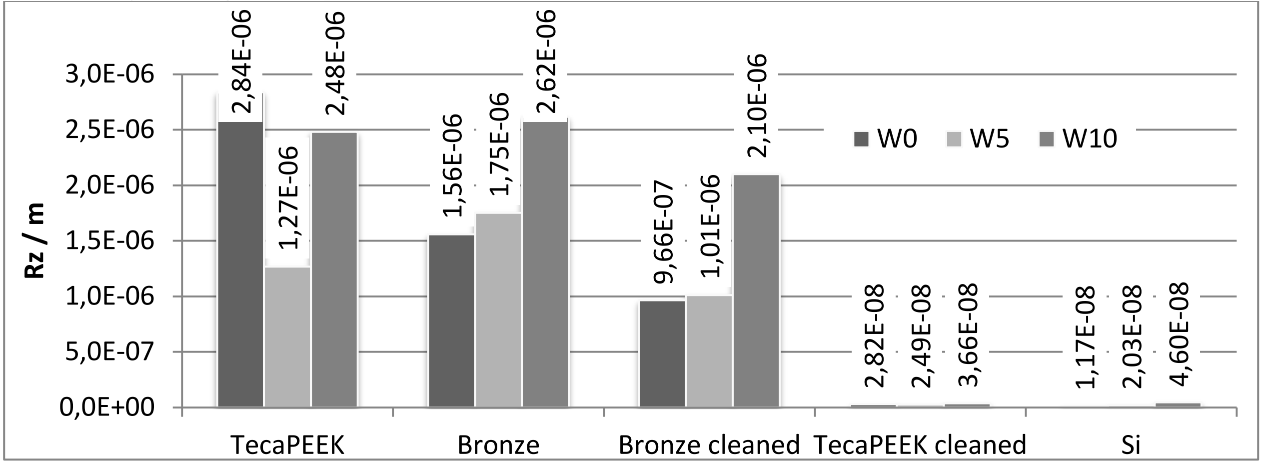


Table 5: surface roughness depth



The values for the arithmetic mean surface roughness are significantly smaller than those measured on the steel surface. This agrees with the facts, that silicon has a very high Young's modulus, high surface hardness and also a very smooth surface in the initial condition of the sample. The measurement of the surface roughness depth is very sensitive to small defects with high magnitude which occur often when scanning the impact area on the silicon surface with the autofocus sensor. This can be due to the behavior of the optical sensor on sharp edges and also due to the transmittance of the silicon oxide. This overestimation was confirmed by scanning the surface with an AFM and a value for Rz of 0,15 µm. For the evaluation of Ra the results from autofocus probe and AFM match.

A correlation of the configuration of the surface oxide layer and mechanical wear could not be seen.

5. CONCLUSION

A setup was presented that allows the experimental simulation of wear of a weight to a spherical contact point to evaluate various material combinations. Furthermore an according testing procedure was developed including cleaning, measurement and data evaluation. In contrast to the actual use of weights, including transport and measurement in different balances, in the experimental setup all contacts take place in only one contact area with no lateral motion of the contact pin. On the one hand this results in an overestimation of the effects on the surface of the weight, but on the other hand the results are very reproducible and also well suited for comparison of material combinations.

For the design of the contact points in storage vessels and automatic load changers different parameters can be used. Given a certain material for the weights, the geometry (radius of spherical pin), and the stiffness of the contacting parts can be chosen. Both theoretical analysis and measurements suggest the use of PTFE and PEEK. While this can be true for storage containers, some additional factors need to be considered such as mechanical stability, contamination of the weight, electrical conductivity. Also especially for soft materials there is a risk of contamination of the contact material with hard particles. To ensure easy to clean surfaces with a high load factor polished contact points are suggested. This is also supported by measurements with polished bronze pins.

The measurements of silicon in combination with TecaPEEK and bronze lead to similar results. In general the wear of the silicon surface is factor ten lower than for steel and a significant difference for various surface oxide layer could not be measured.

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