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Report on STSM „Linking Macroscopic Friction to Microscale Origins: Nanoparticle based Oil Additives"

The 'Short Term Scientific Mission' was carried out in the group of Prof. Graham Cross at Trinity College in Dublin in the time from 11th to 22nd of April, 2016.

During the STSM steel samples subjected to mechanical load were analyzed with regard to their mechanical properties using the nanoindentation equipment available in the laboratory of Prof. Graham Cross. More specifically, the experiments were aimed at clarifying the mode of action and the reasons for effectiveness of an Oil additive (Rewitec) produced by a cooperation partner of the group of Andre Schirmeisen in Gies-sen. The interest in understanding how this additive works is mostly spurred by the stunning results achieved upon application both in laboratory tests (where a reduction of friction by up to 40% can be observed) and in industrial applications like e.g. lubrication of wind turbines (for more details, please see the corresponding proposal to this STSM).

However, at this point, the additive mixture is mostly based on a phenomenological approach. Thus, optimization must mostly be done by trial and error. Evenly important, the uncertainty about the physical and chemical processes at the interface also hampers its more widespread technological application, since the lack of understanding of the interface processes must often be countered by extensive testing for each specific application.

Therefore, the group of Andre Schirmeisen is currently trying to understand the underlying processes related to the use of this additive on a fundamental nanoscale level and the recently concluded STSM was part of an ongoing multi-method strategy to unravel the mechanisms of the additive on a nanoscale level. Current theories are based on the assumption, that nanoparticles present in the additive are grinded within the contact and eventually form a thin film covering the steel surfaces, which might prevent the formation of microscale welding bridges.

The existence of such a film was recently confirmed by XPS measurements and in parallel to this STSM further XPS measurements were performed at the University of Giessen to clarify the chemical interaction between the Si-film and the substrate. Further analysis is aimed at clarification the geometrical behavior of mechanical contacts subjected to the influence of the additives. Using confocal laser microscopy, it was found that the application of the additive does not only reduce friction, but also has a smoothening effect on the surface, an effect which is considered to be diminishing wear and thus prolonging the lifetime of machinery.

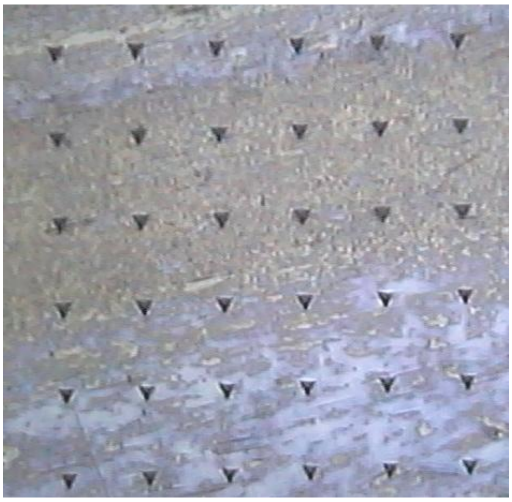
In this context also the mechanical properties of the surface region are important. First of all, the film can be considered as a distinct layer with its own geometrical and mechanical properties. Furthermore, the presence of a layer that reduces friction can in turn also influence the properties of the subsurface region of the samples which are subjected to less strain and temperature increase due to less energy dissipation.

To answer the questions related to these scenarios, we have performed nanoindentation experiments using the 'MTS Nanointender XP' that was available in the group of Prof. Graham Cross. During the measurements, that were assisted by Dr. James Annett, indentation depths up to 2 µm have been chosen, thereby allowing to analyze the surface region as well as subsurface regions. Prior to measurements on steel samples, we have performed calibration measurements on fused Silica, which allows us to quantify the later results.

After that we have analyzed the steel samples, that have been prepared using tribometers under different conditions. More specifically, we have analyzed 3 sets of differently prepared samples. Each set consisted of a sample prepared while the additive was present, whereas a sample prepared without additive was measured for comparison. For each sample, an array of indentations was recorded at two different positions: One inside and one outside the area that was subjected to mechanical stress.

Especially the increased surface roughness in the areas subjected to mechanical stress made it necessary to record a considerable amount of data per sample in order to get reliable results. All in all, data was recorded for 6 steel samples and thus 12 sample positions (More specifically, April 11^{th}-12^{th} was used to set up the equipment and perform first calibrations, while the steel samples were measured from April 13^{th} to 20^{th}, and finally a calibration recheck was done on April 21^{st}).

For the steel samples, 2 sets of have been prepared using the home-made tribometer in Giessen, one in 'pin on disk' configuration and another one in rolling friction configuration. A third sample set was prepared by the tribology center Mannheim using an industrial scale tribometer. All samples have been previously analyzed in Giessen and showed the beneficial influence of the additive. These additional tests should now reveal how different conditions during sample preparation (most importantly with or without adding the additive to the oil bath) reflect in the mechanical properties.



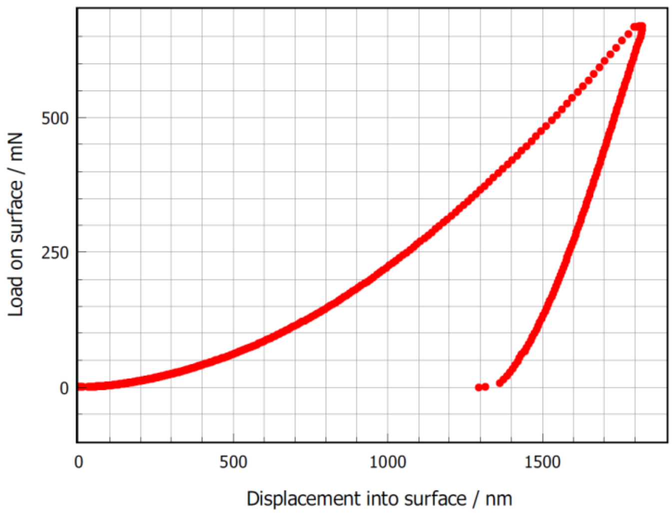


Fig.1) Left: 6x6 Array of nanoindentations in the inner area of a steel sample subjected to mechanical stress, Right: exemplary load vs. displacement curve for one of the indentations of the array.

Fig. 1 shows a typical array of nanoindentations on steel together with an exemplary curve, where the load on the sample is plotted vs. the displacement of the pyramidal diamond indenter. There, the hysteresis of the loading and unloading curve is related to the plastic deformation of the sample. Similar curves have been recorded for all samples described previously. The load vs. displacement curved can now be processed yielding quantitative data like e.g. on depth dependence of the samples elastic moduli or their hardness.

A thorough data analysis is yet still pending but a cursory analysis already indicated that the mechanical stress causes subsurface softening of the sample, that seems to be partially prevented for samples prepared under use of the additive. However, this effect has to be analyzed in more detail for each of the samples under investigation and the analysis would ideally be complemented by transmission electron microscopy measurements as they are planned for the near future.

By only regarding the beginning of the load vs. displacement curves we can also analyze surface effects more specifically. However, special care will be necessary to separate effects of surface roughness and in homogeneities due to different stress conditions (see e.g. Fig. 1), from genuine effects related to the formation of a protective surface layer.

All in all, due to the help of Graham Cross and his co-workers the stay in Dublin has been very fruitful and allowed to gather a large quantity of interesting data. However, since the STSM ended only Friday, 22nd of April with the journey back to Germany, data analysis is still at the very beginning and will commence in the following weeks continuing the good cooperation between Giessen and Dublin.