Data from the research project FYSN21

The data can be found at https://cloud.lucas.lu.se/index.php/s/ooCSrfsg1j02klO

There are 5 experiments done so far, oxidizing the Pd(111) surface in 10 mbar O2 at 500°C for 5, 10, 20, 40 and 80 min respectively. Where to find which data is given in the table below.

The detector images are stored in the corresponding folders. The images cannot be properly shown by the most standard image viewing softwares, but it works with for instance ImageJ, matlab and it is definitely possible to load them with Python, but I do not know how. To simplify the analysis the integrated signal of the oxide reflection (as described in the manual) is also found as text format in the files 221031_Pd111_XX_oxide_signal.cvs. For two of the GIXRD measurement there was a cosmic ray hitting the detector, giving one image a very high intensity. This is fixed buy a "remove outlier" noise reduction tool in the files 221031_Pd111_XX_oxide_signal_noisereduced.cvs. As the sample might (and sometimes has) move during annealing and evacuation of the reactor chamber, the alignment with the oxide reflection may vary. Hence, it is not possible to compare absolute signals between measurements.

The MS data gives a "pressure" of all gases that might be expected to appear in the chamber during the catalysis tests. You are probably mostly interested in the CH4 data, which clearest shows the consumption of the CH4 and hence the reaction. The data need to be treated and normalized a bit before a straight forward comparison can be done. Below, you will find a description of how this was done for the previous course's data. Your data is better so it should be good if you follow the same steps.

| Oxidation time	MS file	GIXRD during oxidation	GIXRD during reaction
5	221031_Pd111_001.txt		
10	221031_Pd111_002.txt		
20	221031_Pd111_003.txt	221031_Pd111_48	221031_Pd111_50
40	221031_Pd111_004.txt	221031_Pd111_60	221031_Pd111_61
80	221031_Pd111_005.txt	221031_Pd111_71	221031_Pd111_72

Treatment of MS data in previous course

Plotting the raw CH4 data you get something like this:
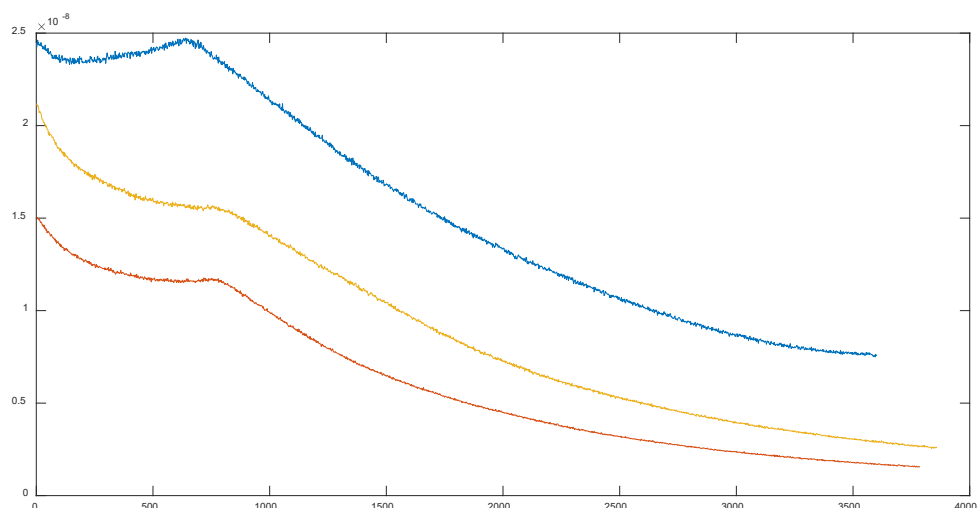
It is very difficult to compare these due to three problems. 1. There is a background signal that differs between the measurements, probably mainly caused by differences in the previous exposure of the MS to gases (for instance that the pump was off one of the nights). 2. The absolute signals cannot be compared, since the leaks from the reaction chamber to the MS chamber was not the same every time. 3. The starting time of the reaction differs from the first to the other experiments.

Starting with the background, we have to assume that if we wait infinitely, all CH4 will have reacted and disappeared. The best would then be to extrapolate the curves until they are flat and subtract the corresponding signal from all values of the curve. This extrapolation is not very straight forward and I will accept if you instead assume that almost all CH4 is converted at the end of the measurement. Hence, you remove the final value of each measurement.
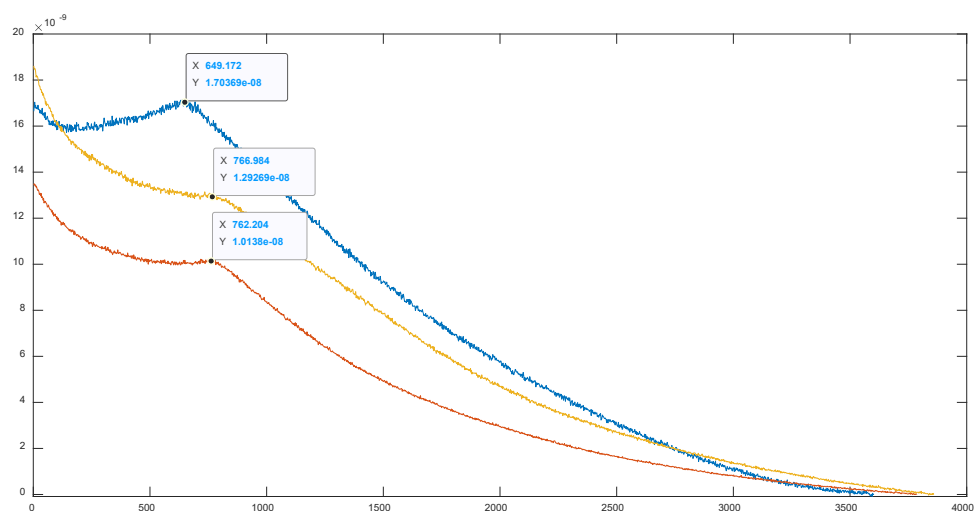
Now normalize this by dividing the signal of each curve with the signal at the start of the reaction. Also, if you multiply by 100, the y axis will tell you how many % of the initial CH4 gas is left.
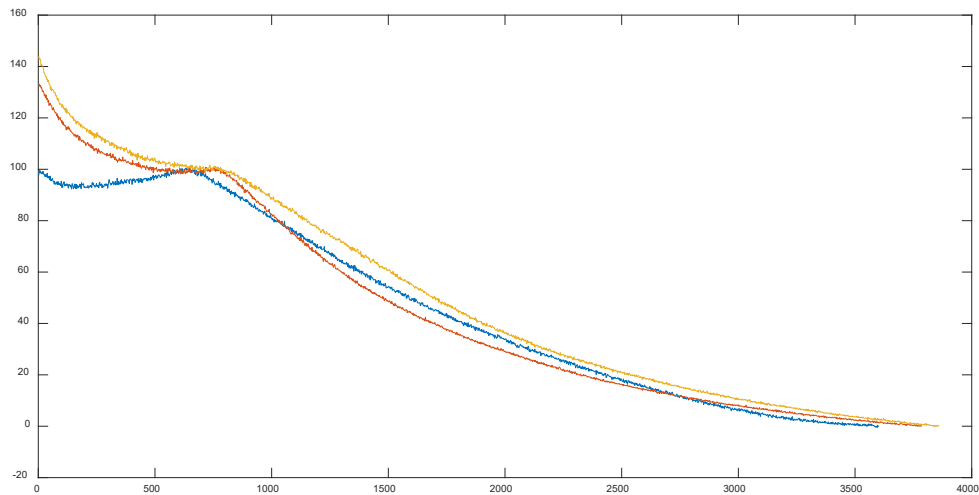
Finally, redefine the time axis such that t=0 corresponds to the point when the heating current have reached 1.4 A and the GIXRD measurements start. This should closely correspond to the point when the curves turn downwards. We then get
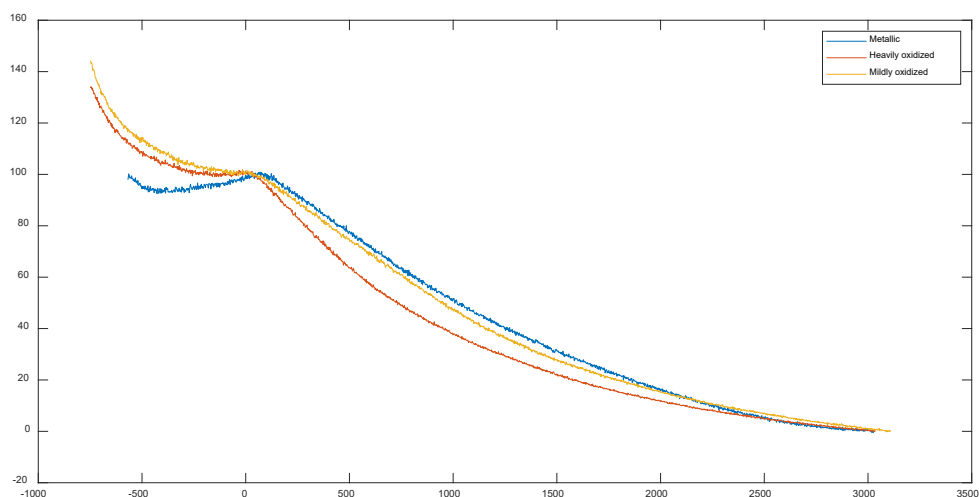
Now it is straight forward to compare the activity in the three experiments.

What happens before t=0 is that the gases that were stuck on the filament of the MS before it was turned on, cause some varying background which slowly stabilizes. Ideally, we should have waited until it was really stable, but we did not have time to do that. This might have some affect on the actual measurement as well, but I don't think it is very significant.

So, the main result is that the thick oxide is more active than the metallic and mildly oxidized surfaces. This is actually contradicting the hypothesis from literature, saying that the activity should go down when the oxide grows too thick.