

TDS Data Processing

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1 Introduction

There are several things we need to understand before we start processing the TDS data. First, H_2 was found not only adsorbed on the front surface of the crystal during beam dosing, but also the back of the crystal. This has been a known issue, but especially stands out in this set of experiments possibly due to the high H_2 beam pressure (80 psi) and long beam exposure time (300s). When dosing, the background pressure of H_2 is at around 8×10^{-8} Torr. It is equivalent to 24 L of H_2 background dose, which is too significant to be ignored. To solve this problem, the Ni sample was prepared in such a way that its front surface was covered by a thick layer of Au particles (QCM frequency loss=250Hz). By doing so, the front face of the crystal should be completely prevented from adsorbing any hydrogen. A TDS spectrum was then collected, which we call *background TDS*. The difference of the regular TDS and the background TDS should be the amount of H_2 desorbed from the front face of the Ni surface. However, this leads to another problem.

When comparing different TDS spectra, the Mass Spec sensitivity has to be taken into account. The easiest way is to normalize all the TDS data to that of the background TDS so that all the TDS spectra are comparable. To do this, we measured the mass spectrometer signal for a series of fixed background pressure of H_2 , allowing us to correct for any day-to-day variations in mass spectrometer sensitivity due to factors such as multiplier gain.

2 Data Processing

2.1 Step One-Obtain the Background TDS and sensitivity

As mentioned above, the background TDS, as shown in Figure 1, was obtained for the sample where its front surface was covered by a thick layer of Au particles. Clearly, a substantial amount of H_2 observed between 200 K and 400 K should be attributed to the surfaces other than the front surface of the crystal.

A sensitivity experiment was performed at the end of the day. The mass spectrometer signal of H_2 was measured at different pressures, as shown in Figure 2. The averaged counts and their corresponding pressures, including the

origin, were fitted using a linear regression model. The slope of the fitted line is the sensitivity of the mass spectrometer on that day. The same procedure was also followed for other TDS experiments as well. Sensitivity experiment is usually done at the end of the day.

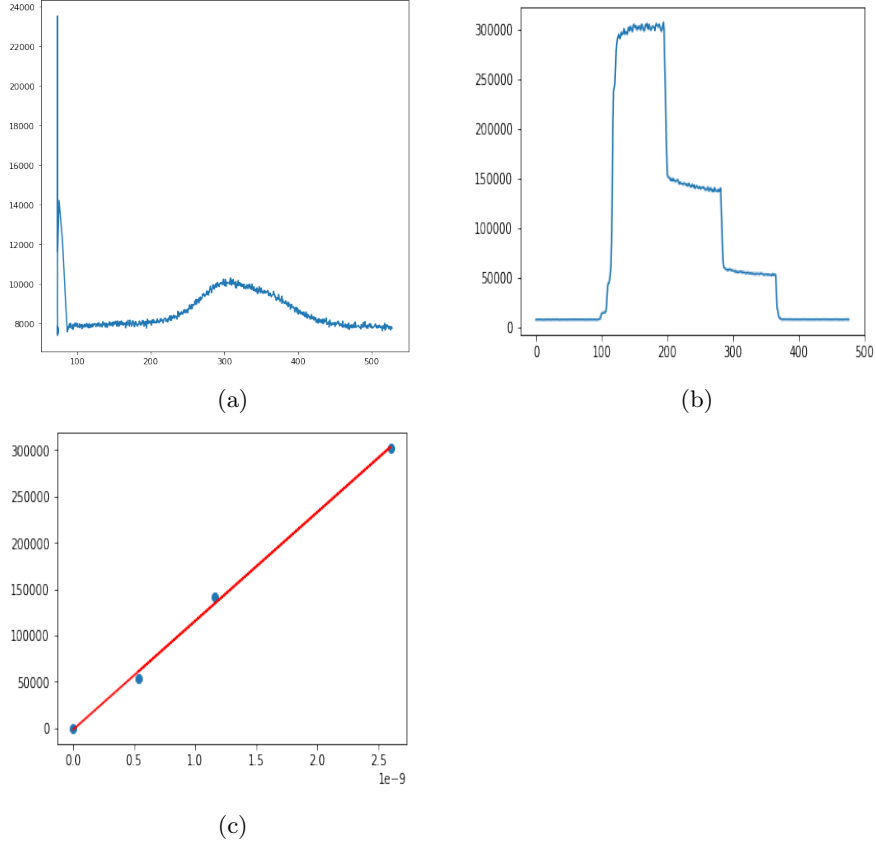


Figure 1: a) H_2 background TDS taken for the Ni crystal whose surface was covered by a thick layer of Au particles. b) H_2 mass spectrometer signal vs. time at different background pressure. c) The fitted linear regression of the averaged counts against their corresponding pressure.

2.2 Step Two-TDS Spectrum Preprocessing

So far, we have done a series of TDS and CIRD experiments to discover the nature of H_2 adsorption on NiAu alloy surface. Each set of experiment is consisted of two TDS spectra, which are a reference TDS taken before CIRD experiment, and a TDS spectra taken after CIRD, a CIRD plot (H_2 counts vs. time), and a sensitivity plot. The collected data on Nov28 is taken as an example, shown in Figure 2.

Before normalizing a TDS spectrum, we preprocess the data first. Shortly after the heating starts during a TDS experiment, the heating filament will release a substantial amount of H_2 , which can be detected by the Mass Spectrometer, as shown in Figure 2a. We then remove this spike by eliminating those datapoints before 95 K as shown in Figure 2b.

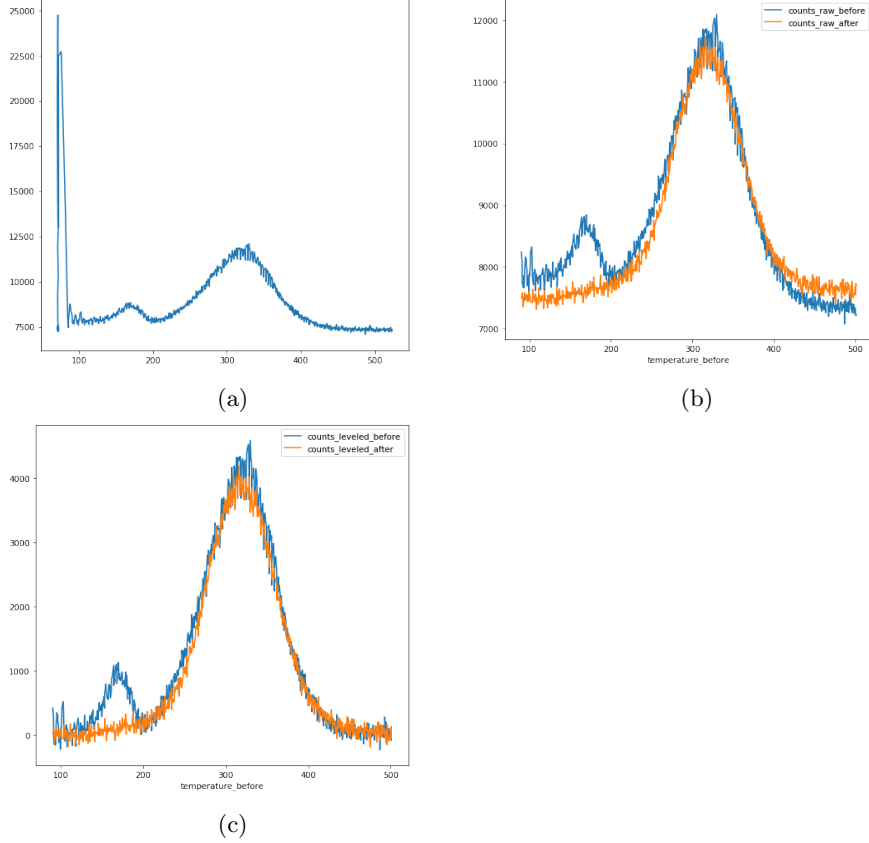


Figure 2: a) a raw TDS spectra taken before CIRD experiment. b) TDS spectra taken before and after CIRD experiment. The spikes at the beginning of TDS experiments have been removed in both spectra.c) Both spectra have been levelled and the baseline has been moved to the x-axis.

When we carefully examine Figure 2b, a problem becomes apparent, *i.e.*, the difference of their baselines, which is due to the residual H_2 during TDS experiments. This effect is especially dominant in CIRD plot. To eliminate this effect, we collect 10 datapoints at the beginning and the end of the spectrum, and calculate the slope of the linear regression of these datapoints, and use it to level the spectrum. In order to make the comparison between TDS spectra easier, we further lower the baseline to 0. Overall, after this adjustment, all the

TDS spectra have the same baseline, *i.e.*, the x-axis, as shown in Figure 2c.

2.3 Step Three-Sensitivity Correction

Now that all the TDS spectra, including the background TDS, have the same baseline, in order to do the background subtraction, we need to correct the sensitivity first, in other words, all TDS spectra should be normalized to the sensitivity of the background TDS. To do this, we use the following equations:

$$\text{Sensitivity Factor} = \frac{\text{Current Sensitivity}}{\text{Background Sensitivity}}$$

$$\text{Counts_Corrected} = \frac{\text{Counts_Original}}{\text{Sensitivity Factor}}$$

Figure 3 shows the difference of two TDS spectra before and after sensitivity correction. The sensitivity for the background TDS is 1.4754×10^{14} , and the sensitivity for the TDS that we are interested in is 1.1723×10^{14} . Because current sensitivity is less than that for the background TDS, it means mass spectrometer is less sensitive as compared to when the background was performed. As a result, after the sensitivity correction, the counts become higher. Note that the baseline is still unchanged after sensitivity correction because the baseline is at the x-axis, *i.e.*, 0.

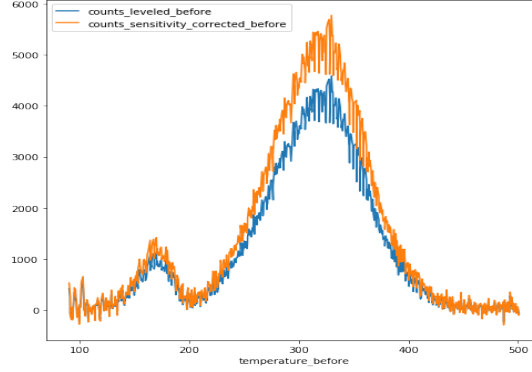


Figure 3: TDS spectra before and after sensitivity correction.

2.4 Step Four-Background Subtraction

Up to this point, all the TDS spectra, including background TDS, are leveled, and have the same baseline and sensitivity, so they are ready to do the background subtraction. Figure 4 shows the difference of TDS spectra before and after background subtraction for the TDS experiments done on Nov28.

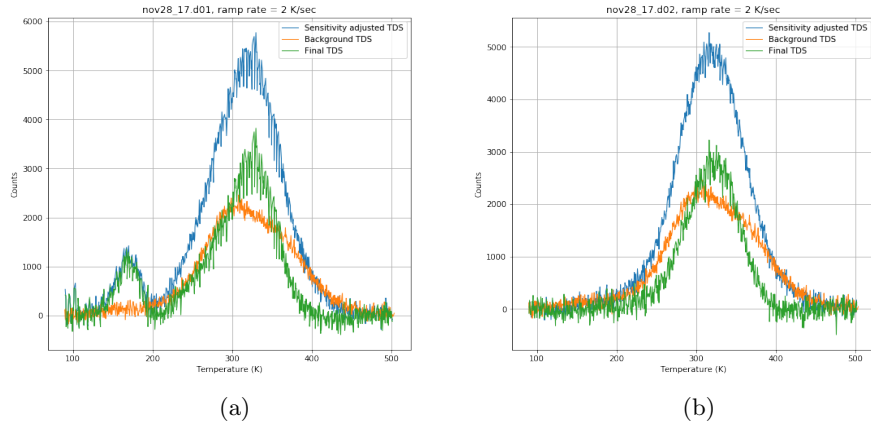


Figure 4: H₂ TDS spectra on AuNi alloy surface taken (a) before CIRD experiment (b) after CIRD experiment.

3 Notes

The whole process was automated with a program written in python. The program will output a txt file that contains all the processed data and can be imported into Igor for data visualization.