Temperature Programmed Reduction

TPR with the Hiden *QIC* Series

Temperature Programmed Reduction

Temperature programmed reduction (TPR) experiments are a very common catalyst characterisation technique. TPR involves linearly heating a sample under a reducing atmosphere such as H_2 or CH_4 and measuring the consumption of the reducing gas and the reduction products formed. The TPR profile obtained from this type of experiment gives information about the reducibility of the sample.

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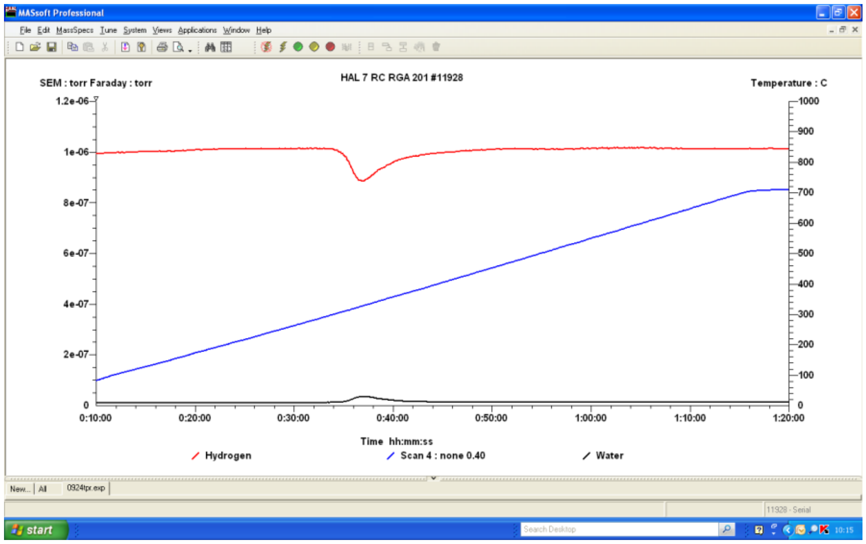

Figure 1 Typical TPR profile

QIC Series

The QIC series gas analysers from Hiden have a number of features making them ideal for TPR experiments. The HPR-20 *QIC* is shown in Figure 2.

Figure 2 HPR-20 *QIC*

QIC series gas analysers feature a dual rotary pump sampling system to maximise the pumping efficiency of the gas being sampled. The *QIC* Inlet has a high throughput of gas into the sampling system, *ca.* 16 ml/min, which gives excellent response times and minimises any dead volumes in the experimental setup. The dual rotary pump design ensures that the majority of the sample gas is pumped by the sample bypass rotary pump independently of the mass spectrometer chamber turbo pump. This is especially important for TPR experiments where relatively high levels of light gases such as H_2 are used. Without this pumping system there will be a build up of H_2 in the mass spectrometer chamber as a turbo pump cannot easily remove high levels of H_2. This can lead to instability in the MS due to a varying H_2 background signal. As TPR experiments often show only a small change in H_2 signal during the reduction it is important to have a stable baseline in order to clearly measure the reduction properties.

The *QIC* series also allows the integration of external devices such as temperature inputs, see Figure 3, and control of devices such as mass flow controllers making them ideal for integration into any experimental setup.

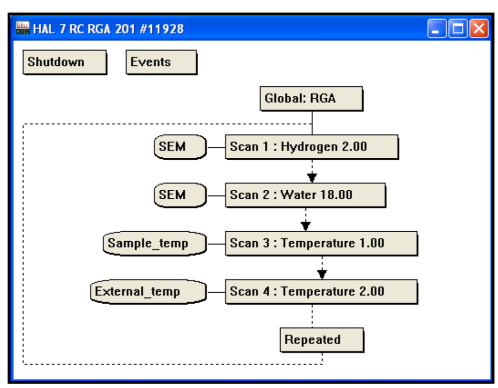

Figure 3 MASsoft Setup with Temperature Inputs

TPR Data

The following data shows a number of TPR profiles. All the experiments were performed using a Hiden CATLAB microreactor system coupled to a *QIC* series gas analyser.

Figure 4 shows the TPR profile of a plasma modified Ni/MgO catalyst.

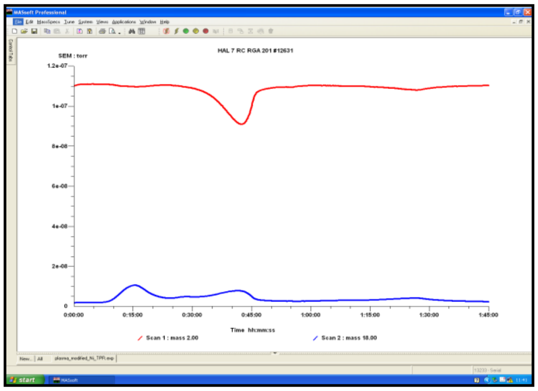

Figure 4 TPR of Plasma modified Ni/MgO Catalyst

Figure 5 shows the TPR profile of a 5% Ni/MgO catalyst. Here the H_2 consumption is low, demonstrating the importance of a stable and sensitive mass spectrometer.

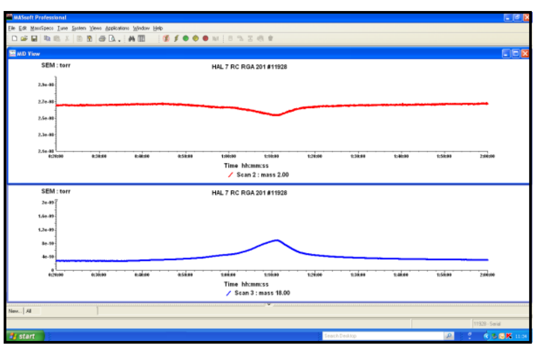

Figure 5 TPR of 5% Ni/MgO Catalyst

Figure 6 shows a TPR profile using CH_4 as the reducing gas on a Ni/SiO_2 catalyst. Here CH_4 is consumed during the reaction forming H_2 as one of the products.

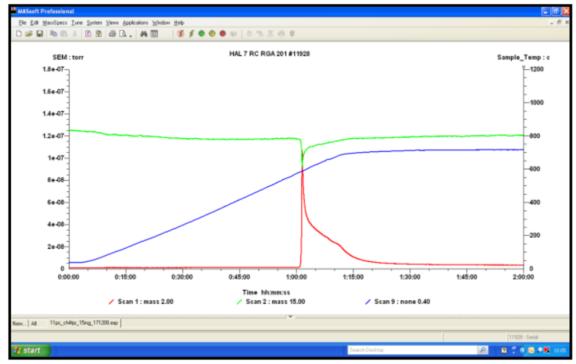

Figure 6 CH_4 TPR Ni/SiO_2 Catalyst

This figure demonstrates the advantage of using a mass spectrometer over other detection techniques such as thermal conductivity detectors (TCDs). The mass spectrometer can detect and identify a number of components simultaneously whereas a TCD can only measure changes is gas composition and provides no information on its identity.

Figure 7 shows the the TPR profile of a CuO sample.

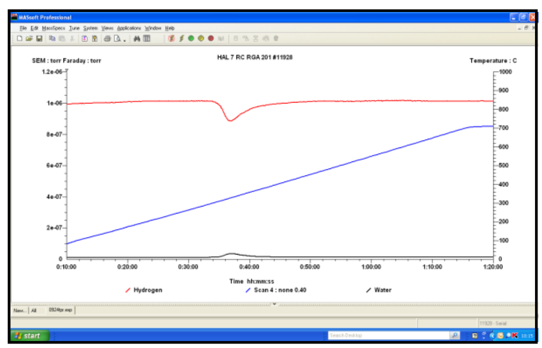

Figure 7 TPR of CuO sample

Conclusion

The Hiden QIC series of gas analysers shows excellent stability and sensitivity for TPR experiments.