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TPD Workstation

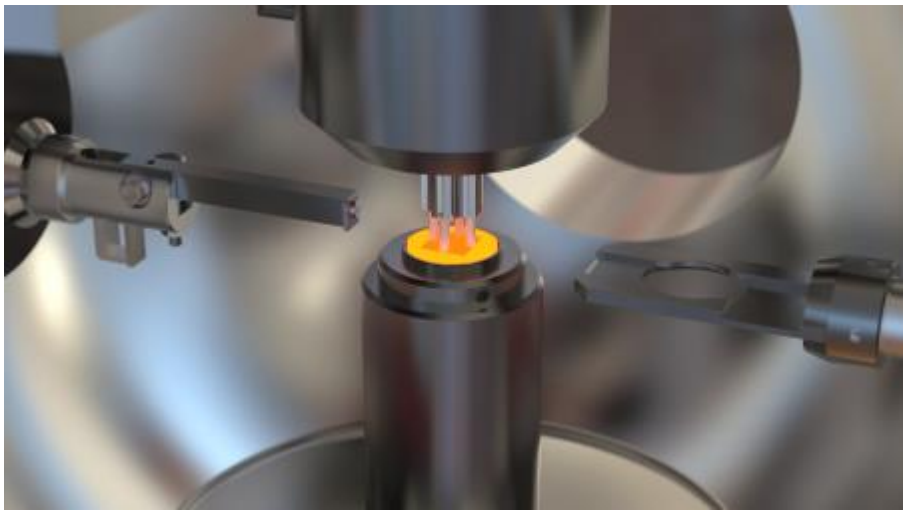
Analysis of Hydrogen Isotopes in Metals by TPD/TDS

Hiden TPD Workstation

Introduction

The detection of hydrogen isotopes and helium in metals has a wide range of applications from hydrogen embrittlement studies to tritium retention in fusion reactor wall tiles [1, 2]. A successful method for investigating the amount and mobility of these species is temperature programmed desorption (TPD), also known as thermal desorption spectrometry (TDS) or thermal desorption analysis (TDA). Analysis by TPD involves positioning the sample in an Ultra High Vacuum (UHV) chamber and heating the samples at different linear ramp rates while collecting the desorption spectra using a quadrupole mass spectrometer. The Hiden TPD Workstation is a complete experimental workstation designed for this application and is optimised to obtain the maximum sensitivity for desorption of hydrogen isotopes and helium from metals.

1. Phys. Scr. **T167** (2016) 14074
2. Phys. Scr. **T167** (2016) 14075



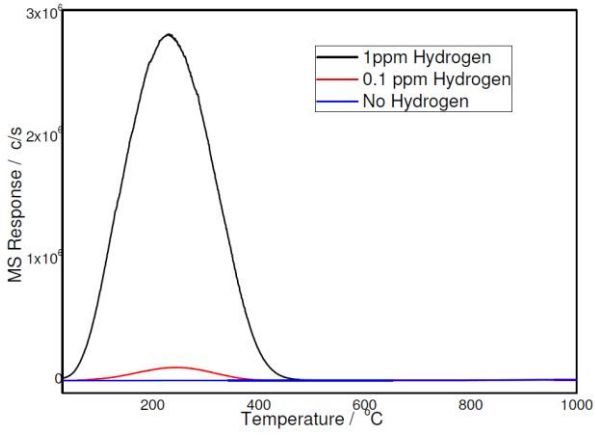
TPD Sample under analysis

Example Data

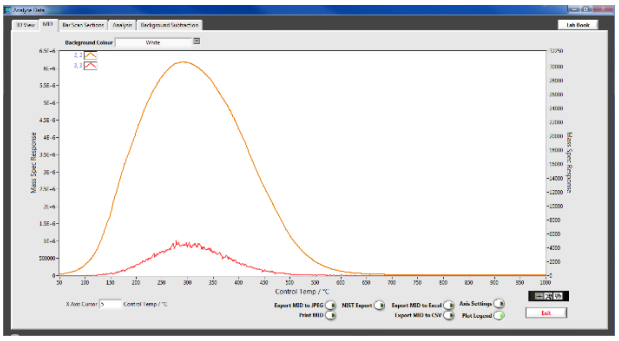
The Hiden TPD Workstation has several features to optimize the sensitivity of the system for low level desorption products. These include:

- No sample holder – only the sample enters the UHV chamber meaning no outgassing components other than the sample.
- High sensitivity triple filter PIC quadrupole mass spectrometer.
- Close coupling between the sample and mass spectrometer for maximum sensitivity and optimum desorption profile.
- Cooled mass spectrometer shroud to minimize background contributions.

The data below shows typical sensitivity of the system. Here stainless steel samples impregnated with hydrogen were heated to 1000°C. Three samples were run as a comparison. Sample 1 contained no hydrogen, sample 2 contained 0.1 ppm hydrogen and sample 3, 1 ppm hydrogen. The data clearly shows that 0.1 ppm hydrogen can be easily detected and that the background levels make no contribution to these signals.

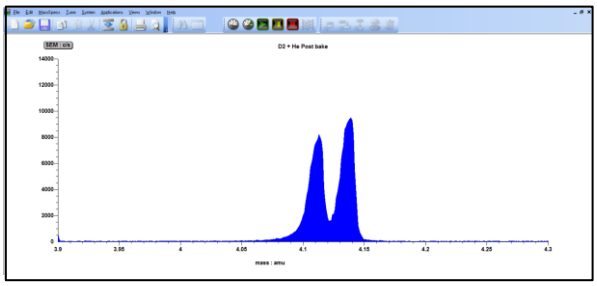


The data below shows the same 1 ppm hydrogen impregnated sample as above and here shows the comparison of the H_2 (Y1 axis) with the desorption of HD (Y2 axis). The data shows the sensitivity of the system as it detects the HD at levels due to the natural abundance of deuterium in hydrogen.

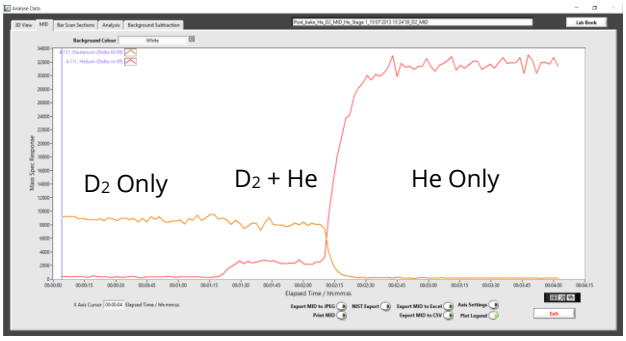


D_2/He Desorption

For more advanced applications that require detection of species with the same nominal mass the Hiden TPD Workstation can be configured with a 9 mm quadrupole analyser. The increased quadrupole rod diameter compared with the standard 6 mm rod size allows the resolution of the instrument to be increase allowing separation of two species with very close mass to change ratios. The data below shows the resolution of He (m/z 4.0026) and D_2 (m/z 4.0282), a difference of 0.0256 amu.



Using the system with these settings allows He and D_2 to be analyses real time for accurate determination of He and D_2 in the sample. The data below shows the real time separation of these species.



Conclusions

The Hiden TPD Workstation is ideal for examining hydrogen in metals and its uses can also be expanded to resolving the detection of species with the same mass such as He/D_2. Additionally any other gases within the mass range of the instrument can be detected simultaneously with the species described here. The TPD Workstation is easy to use and offers high sensitivity and repeatability.