**HELIUM BEHAVIOR IN METALS CHARACTERIZED BY THERMAL HELIUM DESORPTION SPECTROSCOPY—**S. C. Glade and B. D. Wirth (University of California, Berkeley) and H. Schut (Interfaculty Reactor Institute, Delft University of Technology, Delft, The Netherlands)

## OBJECTIVE

To construct and operate a thermal helium desorption spectroscopy experimental characterization system for use in analyzing helium behavior in iron and related fusion materials.

## SUMMARY

A facility to perform thermal helium desorption spectroscopy is under construction at the University of California, Berkeley. The facility is scheduled to be validated and in operation in early June 2005. Experiments are currently being designed to provide data on helium diffusion, trapping mechanisms and energetics in iron and iron alloys.

## PROGRESS AND STATUS

### Introduction

The behavior of helium in metals must be understood for future use in fusion applications. In fusion reactors, the helium concentration in metals increases by direct implantation of helium and by nuclear transmutation reactions. Helium has extremely low solubility in most metallic alloys and interacts (traps) strongly with preexisting and radiation produced microstructural defects. At high concentrations, helium precipitates to form helium bubbles which can cause high-temperature intergranular embrittlement and swelling. Of particular interest is the behavior of helium in iron, for which there is very little experimental data [1–3].

A thermal helium desorption spectrometer (THDS) is under construction at the University of California, Berkeley. Its design is based on THDS systems at the Delft University of Technology. The THDS method and information that can be obtained from analyses of the desorption spectra are presented in this progress report.

### Experimental Design

Conceptually, thermal helium desorption spectroscopy is simple: heat the material of interest and measure the amount of helium released. The design requirements for a thermal helium desorption spectrometer are presented in Refs. [4–6], and an example of an operating system is provided in Ref. [7]. A schematic of the system at U.C. Berkeley is shown in Fig. 1.

#### Vacuum System

The THDS system is constructed with commercial stainless steel vacuum components and all-metal valves. The total chamber volume is approximately 2.5 L, pumped with two turbo-drag pumps backed by roughing pumps. The two turbo-drag pump setup allows the mass spectrometer to always remain under vacuum, with only the sample holder and heating system brought to atmospheric pressure for sample changes. Bayard-Albert type hot cathode ionization gauges are used for pressure measurement, with the expected vacuum level of the system to be $10^{-8}$ Pa ($7.5\times10^{-11}$ torr). A liquid nitrogen cooling coil in the sample chamber (not shown in Fig. 1) will be used to prevent heating of chamber walls as well as to condense gases other than He that are desorbed from the sample.

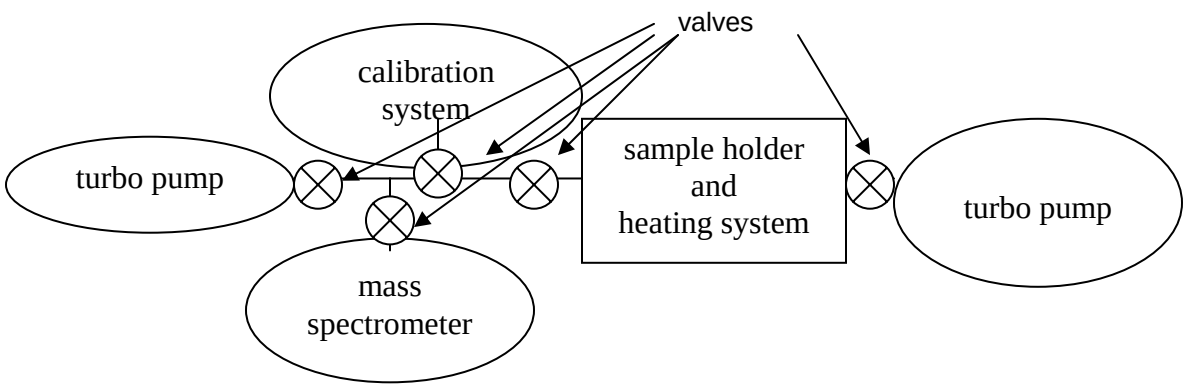


**Fig. 1. Schematic of thermal helium desorption spectrometer.**

Helium Detection

Helium detection in the THDS is preformed with a mass spectrometer equipped with a Channeltron type electron multiplier. A linear relationship exists between ion current in the mass spectrometer and helium partial pressure:

$$I(A) \propto P_{He}(torr)\,. \qquad (1)$$

The helium desorption rate from a sample is described by:

$$L(t) = \frac{dN}{dt} = \frac{dP(t)}{dt} + \frac{S}{V}P(t) = \frac{dP(t)}{dt} + \frac{P(t)}{\tau}\,, \qquad (2)$$

where *L(t)* is the helium desorption rate,*N* is the number of helium atoms,*P(t)* is the partial pressure of helium,*S* is the pumping speed (L $s^{-1}$),*V* is the chamber volume (L), and $\tau$ = *V/S*, the pumping time constant (s).

The THDS system can operate in two modes, either a static or a dynamic mode. In the static mode, the $P(t)/\tau$ term in Eq. (2) is minimized, with no pumping on the sample holder chamber. In the dynamic mode, the $dP(t)/dt$ term in Eq. (2) is minimized, and $\tau$ is adjusted by the valve to the turbo-drag pump. We plan to operate the THDS system in the dynamic mode.

The mass spectrometer will be calibrated by releasing a known small amount of He into the system to determine a calibration factor:

$$Q = {\#He\ atoms}/{I} \qquad (3)$$

Sample Heating

The samples will be heated radiatively by a resistively (150 A, 20 V) heated tungsten filament. A type C thermocouple is used for temperature measurement. Proportional integral derivative (PID) temperature control will be implemented with LabVIEW™. Different heating schedules can be programmed with the system, with either linear heating or linear-step heating (i.e., heating to T1, cooling to room temperature, heating to T1 + 50 K, cooling to room temperature; this is continued in $\Delta$T steps until the maximum

desired temperature is reached) used initially. LabVIEW™ is also used for recording all system data during a desorption experiment (temperature, pressure, He partial pressure, and time).

**Data Analysis**

The first-order rate of He desorption from a sample is given by:

$$\frac{dN}{dt} = -N\nu \exp\left(\frac{-Q}{kT}\right), \tag{4}$$

where $N$ is the number of helium atoms, $Q$ is the activation enthalpy, $\nu$ is the attempt frequency, $T$ is temperature, and $k$ is Boltzmann's constant. With a linear heating rate, the temperature at which the desorption rate is maximum is:

$$-\frac{Q}{kT_{\max}^2} = \frac{\nu}{\beta}\exp\left(-\frac{Q}{kT_{\max}}\right), \tag{5}$$

where $T_{max}$ is the temperature at which the desorption rate is a maximum and $\beta$ is the heating rate. Eq. 5 is used to calculate the activation enthalpy, $Q$. The attempt frequency, $\nu$, is usually taken as the Debye frequency, $1\times10^{13}$ $s^{-1}$, which is on the order of the atomic vibration frequency in solids.

Linear-step heating experiments can provide further information about helium release mechanisms. By considering the first part of the release curve where the change in $N$ is very small, the slope of a release curve, when plotted in an Arrhenius plot, gives the activation enthalpy, $Q$. The attempt frequency, $\nu$, can also be calculated, instead of assuming a value. A calculated attempt frequency lower than the expected Debye frequency value indicates multi-step activated processes in which first-order reaction kinetics do not apply. Examples of both linear heating and linear-step heating THDS experiments are presented in Ref. [8].

We anticipate multiple He interactions govern the He desorption, which make data analysis more complicated than simple first-order kinetics. The planned THDS experiments will be closely coupled to multiscale simulation models currently being developed.

**Progress**

The procurement of all required equipment for the THDS system is complete. The vacuum system is assembled, and vacuum levels of $10^{-8}$ torr have been demonstrated. The next planned steps to prepare the system for experiments include attaching the mass spectrometer, testing the sample heater, and installing the calibration system.

**Future Work**

The THDS system is planned for initial operation by the beginning of June 2005. We will soon begin helium implanting iron specimens with an ion gun for use in system validation. After the system is operating and validated, we will begin experiments to provide data on helium diffusion trapping mechanisms and energetics in iron.

**Acknowledgments**

This work has been supported by the Office of Fusion Energy Sciences, U.S. Department of Energy, under Grant DE-FG02-04ER54750.

**References**

[1] R. Vassen, H. Trinkaus, and P. Jung, Phys. Rev. B 44 (1991) 4206.
[2] K. Morishita, R. Sugano, and B. D. Wirth, J. Nuc. Mater. 323 (2003) 243.
[3] K. Morishita, R. Sugano, B. D. Wirth, and T. Diaz de la Rubia, Nuc. Inst. Meth. Phys. Res. B 202 (2003) 76.
[4] P. A. Redhead, Vacuum (1962) 203.
[5] A. A. van Gorkum and E. V. Kornelsen, Vacuum 31 (1981) 89.
[6] E. V. Kornelsen and A. A. van Gorkum, Vacuum 31 (1981) 99.
[7] E. Kautto, O. P. Kahkonen, J. Kuhalainen, M. Manninen, Nuc. Inst. Meth. Phys. Res. B 103 (1995) 376.
[8] P. M. G. Damen, A. van Veen, F. Labohm, H. Schut, and M. A. van Huis, J. Nuc. Mater. 306 (2002) 180.