

New Folder Name Outgassing Procedure

Procedure for Measuring H₂ Outgassing from Steel Coupons

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1. Scope:

The procedure described here covers hydrogen outgassing measurements from samples consisting of steel sheet (henceforth called coupons). These outgassing measurements are used to qualify/screen steel coils, or to validate various treatments of the steel, e. g. cleaning.

2. Personnel:

Outgassing measurement should be performed by experienced and trustworthy personnel.

3. Equipment and materials:

- a. Outgassing measuring equipment, with the rough outline shown in Fig. 1.
- b. Liquid nitrogen.

4. H₂ measurement procedure:

- a. Place coupons inside test chamber, start pumping.
- b. Bake coupons at 250°C, for 36 hours¹
- c. After turning the heating off, let the system cool and the outgassing settle for 3 days.
- d. Close valve V1, in the morning, and let hydrogen accumulate for several hours.² Record valve closure time.
- e. Fill the liquid nitrogen trap (about one hour before the outgassing measurements).
- f. Just before starting the outgassing measurement, close V2 on the calibrated leak, to accumulate a known amount of hydrogen. Record valve closure time.

¹ That is the heating will be kept on for 36 hours.

² Accumulation for the first measurement in a series should be 6 hours. The following measurements are best carried out on a daily base, with ~24 h accumulation times.

- g. Outgassing measurement:
- Close V3, open V1.
 - Start RGA scan (pressure versus time mode, 80 s span, 10 ms dwell time).
 - Open V3, record time. This time minus the chamber closure time gives the accumulation time for hydrogen from the sample, t_s .
 - Save the pressure versus time plot recorded by the RGA, record the peak pressure reading p_s .
- h. Calibration:
- Close V3, open V2, with V1 open.
 - Start RGA scan (pressure versus time mode, 80 s span, 10 ms dwell time).
 - Open V3. This time minus the leak valve closure time (Point f) gives the accumulation time for hydrogen from the leak, t_l .
 - Save the pressure versus time plot recorded by the RGA, record the peak pressure reading p_l .
- i. Close V1, to accumulate hydrogen from the sample for the next measurement, record the time.
- j. Calculate the hydrogen outgassing from the sample, j_s :

$$j_s = \frac{p_s}{p_l} \cdot \frac{t_l}{t_s} \cdot \frac{r_l}{A}$$

where r_l is the flow rate from the calibrated hydrogen leak, and A is the total area of the sample.

Note:

Three outgassing measurements will be performed for each sample, in order to ascertain that the outgassing rate is roughly constant with time. In case of serious doubt (e. g. an apparent upward trend of more than 25%), a fourth measurement will be carried out.

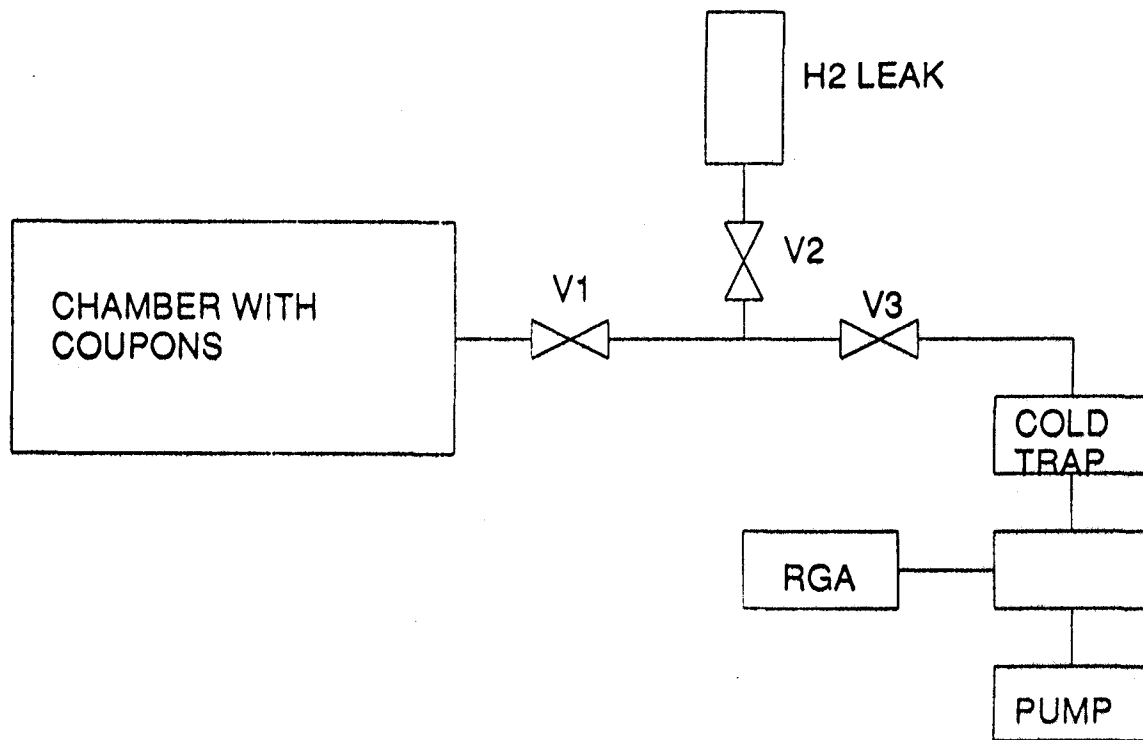


Fig. 1. Coupon outgassing measurement arrangement.