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EQUIPMENT FOR STUDYING HOT CORROSION PROCESSES IN DYNAMIC GAS MIXTURES CONTAINING WATER VAPOUR

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ABSTRACT

A Setaram unit including a symmetric oven and a MTB 10/8 microbalance was chosen as a base equipment in order to reduce the impulse variations frequently occurring in dynamic regimes. The thermobalance and its surroundings were thoroughly thermostated owing to water vapour. To pull up and let down the sample quickly without damage for the torsion strip, a telehandling air-tight apparatus with visual control has been worked out, allowing to make optional the connection between the suspension chain and the microbalance beam.

The equipment here described has been conceived for hot corrosion kinetic studies (round 1000°C) concerning low weight samples (10-100mg) in such gaseous flows as X/H_2O containing air, oxygen and hydrogen in the presence of water vapour (1 - 75 %).

SPECIFIC REQUIREMENTS

1 - Kinetics of gas-solid reactions make necessary to follow weight changes as a function of time, while keeping constant experimental parameters related to:

a - the sample environment, such as the temperature and composition of the gaseous mixture. The very beginning of the reaction, on the other hand, must be easily located.

b - the response of the weighing device which transforms mass change into a suitable electric signal.

2 - The presence of water vapour implies that the whole apparatus be heated, including the weighing device.

3 - The circulation of the gas flow results in disturbances which require an adequate experimental configuration for reducing these perturbations.

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CONCEPTION OF THE EQUIPMENT

The experimental device represented in figure 1 includes a classical equipment for the production and regulation (parts A and B) of water vapour containing gas mixtures of well defined composition. Part C, by far the most important, is specially concerned with the reactor and the microbalance which are both symmetrical.

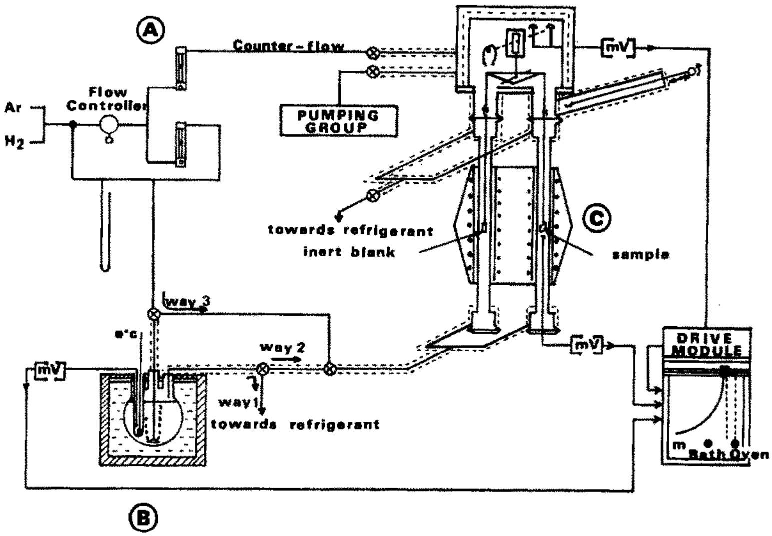

Fig. 1 - Experimental device.

The reactor - oven unit

The disturbances generated by the gas flow near the sample can be overcome by means of two paralell reaction tubes ; one of them contains the sample and the other an inert blank connected by an identical platinum chain to the microbalance. They are both strictly heated in the same conditions.
The refractory tubes, on the other hand, are rigidly locked with the oven and can be moved together in order to raise the sample temperature as quickly as possible.

The microbalance

Tests conducted with a non symmetric balance invariably result in many errors, as it is necessary to create a vacuum through the apparatus, then to come to normal pressure with the carrying gas (air, oxygen and hydrogen) before introducing water vapour. In such conditions, the homogeneity of the reacting gases is rather long to take place, so that the gravimetric results may be deeply affected at the beginning ot the reaction.

This is the reason we have chosen a symmetric device and, in particular, a Setaram MTB 10/8 microbalance which may be thermostated without damage at about 85-90°C by means of a heating muff.

The zero time definition

Before the thermal equilibrium of the reaction zone is reached, the sample must be maintained out of this working zone to achieve the reaction under isothermal conditions. For the same reason, it is also necessary to drop the sample into the isothermal zone and then to record the signal corresponding to its mass.

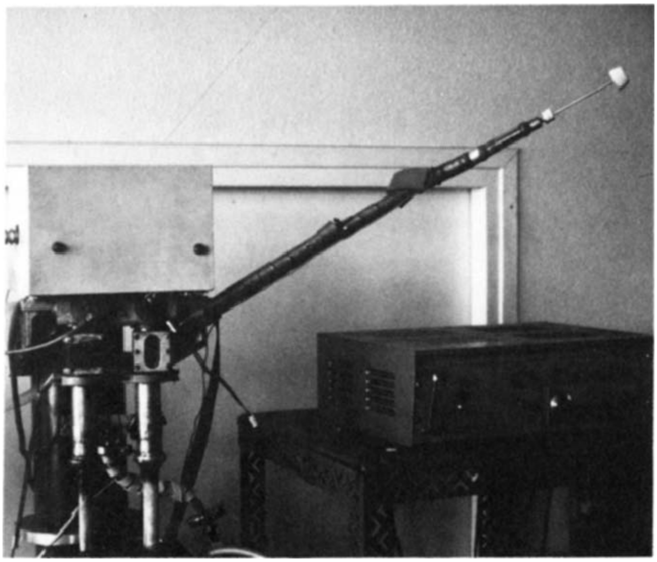

Fig. 2 : General view of the apparatus.

A simple way would consist in rising the suspension chain by means of a lateral opening, but this is not practicable because the balance torsion strip is too brittle to support even moderate stresses. Therefore, we had to conceive a gas-tight device in order to make optional the junction between the chain and the balance beam. Such an apparatus, which is described in the next section, allows one to pull up and let down the sample quickly without any damage to the torsion strip, and to fix with precision the very beginning of the reaction.

THE DRIVE SYSTEM OF CONNECTION BETWEEN THE SUSPENSION CHAIN AND THE BALANCE BEAM

This system, which is located between the microbalance and the refractory tubes, is composed of two identical parts consisting of a stainless steel plate and a side pyrex glass tube (fig. 2).

The plate

Its first function is to realize a quick junction between the weighing gear and the reaction tube by means of a Leybold type connection (fig. 3).

The second is to guide and visually control the connection between a rod hanging from the beam and the suspension chain bearing the sample. This can be done respectively by an curved transversal axis which allows the centring and sliding of the chain, and by a pyrex glass viewing port.

The side tube

It contains the telehandling gas-tight device which is essentially composed of a stainless rod, 4 mm in diameter, ending in a crook towards the balance (fig. 4). By acting on the outside part of the rod, this crook may be moved in

* translation along the tube axis,
* rotation along the rod axis and
* inclination of a few degrees from the tube axis,

due to stainless bellows joining the glass tube and the terminal part which contains a series of silicon polymer ring joints to make it tight.

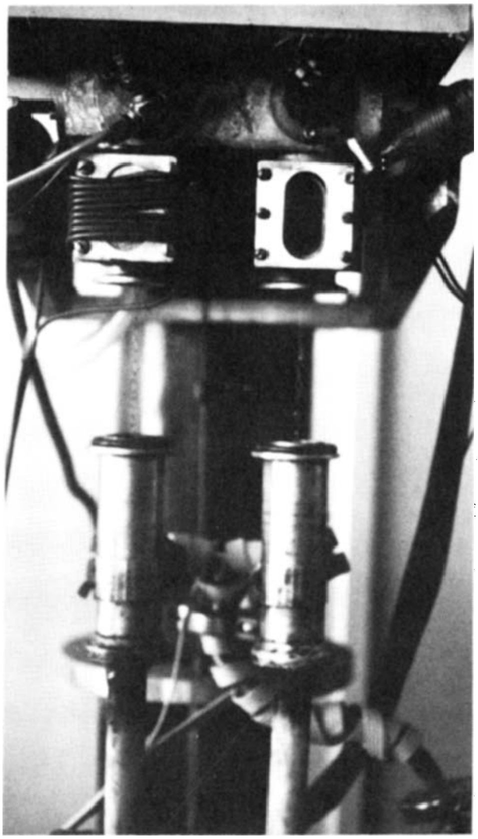

Fig. 3 : Details of the plate.

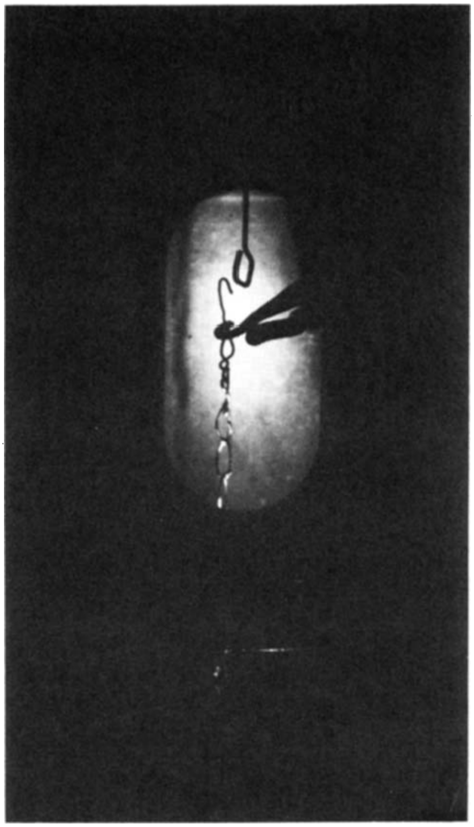

Fig. 4 : Telehandling device. Details.

Some detail of the manipulations that can be made has been illustrated in figures 4 and 5.

It is worth nothing finally that this device, as well as the whole equipment, must be held at temperatures higher than that of the gas mixture condensation by means of warm coiled ribbons.

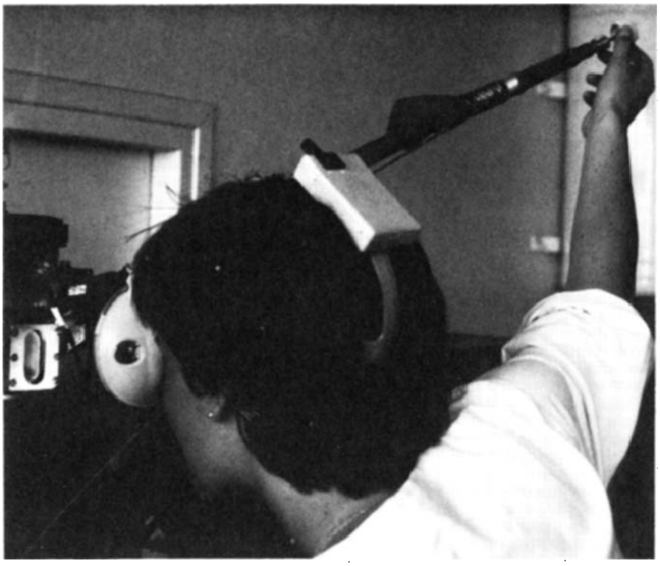

Fig. 5 : Telehandling device. Manipulation.

CONCLUSION

The best advantage of the above equipment is to get accurate and reliable experimental results from a kinetic point of view, especially by locating with precision the very beginning of any reaction at high temperature (1, 2, 3). Indeed, the uncertainty in determining the zero time is of the order of a few seconds, that is the time normally required for taking down the sample and achieving the junction to the balance.

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