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DSC-Instrumentation

MAGNETIC COUPLING FOR A MICROBALANCE

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Balances utilising the principle of free magnetic suspension can be very useful for thermogravimetric experiments, where aggressive atmospheres and high pressures are applied. Magnetic coupling systems with automatically controlled attraction can be used for direct conversion of weight into an electric signal or for the separation of object and beam balance. The magnetic suspension balance is described and a new versatile magnetic coupling system is presented. Due to its low weight, this coupling can be combined with a microbalance.

Introduction

Thermogravimetric experiments are important to scientific investigation and quality judgement of many materials. Thermogravimetric systems generally consist of a balance, a furnace and an equipment to adjust the atmospheric pressure inside the reaction chamber. To extend the range of application, the sample, which is to be examined, can be encoupled to the balance via a magnetic coupling without any mechanical guidance. The sample then can be enclosed inside a hermetically sealed autoclave, where high pressure and aggressive media may be applied. On the other hand, ultra high vacuum inside the reaction chamber will not be influenced by the balance. This was suggested by Gast in 1959 and, according to this principle, many balances for application in the field of thermal analyses have been devised [1, 2].

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Magnetic systems for weighing purposes

The principle of coupling the sample to the balance by magnetic attraction is shown in Fig. 1.

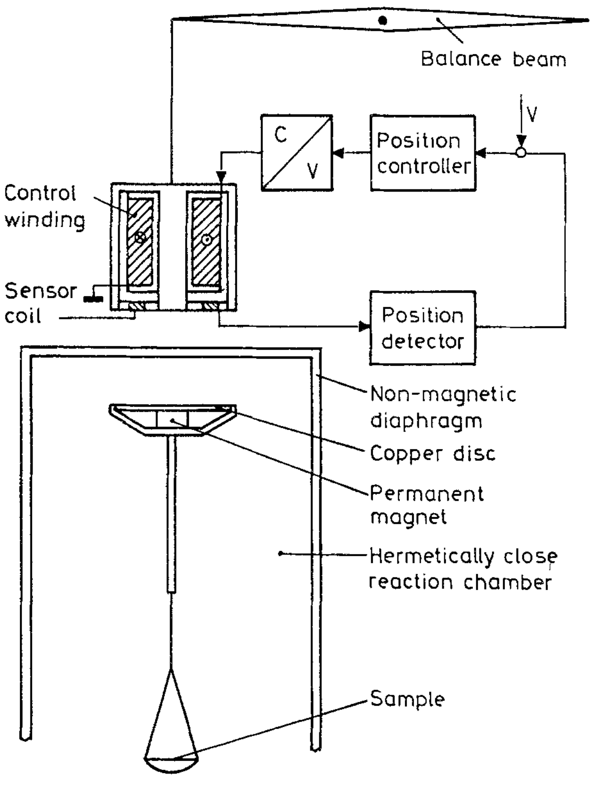

Fig. 1 Schematic drawing of suspension balance with supporting beam balance

A permanent magnet, which is attached to the sample, is kept in floating state by the attracting force of an electromagnet. The floating state is maintained by a position control circuit, consisting of an inductive vertical position detector, a controller and a control winding to generate the attracting force. After comparison with a reference voltage, the position signal is fed to the input of the controller. The loop is completed by a voltage-to-current-converter to force the compensation current through the control winding. A shell of mild steel is provided to improve the efficiency of the regulating unit and to minimize the magnetic stray flux. An insulating, non-magnetic diaphragm separates the described parts.

If sturdy structure of the measuring system is most important, the magnetic suspension may be also used as an alternative to the beam balance. In Fig. 2, an example for a balance utilizing the free magnetic suspension for direct conversion of weight into an electric signal is given. An electromagnet attracts a permanent magnet, which supports the balance pan. The position control circuit consists of a light barrier and an integral controller. The compensation current depends on the weight of the suspended mass and can be measured.

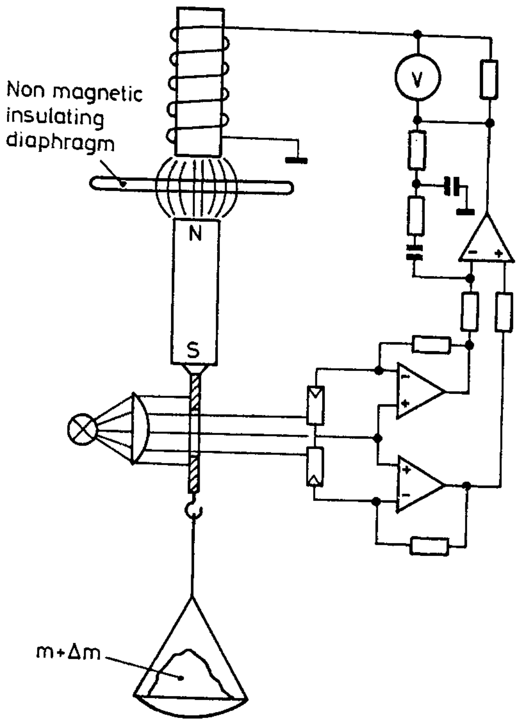

Fig. 2 Directly weighing suspension balance with opto-electronic position sensor

Contrary to the beam balance, the dead weight of the suspended magnet and pan cannot be compensated by any counterweight. The corresponding offset makes this principle less suitable for weighing small masses with an accuracy better than 10 micrograms.

A balance for high pressure application, which is based on this idea, is currently being developed [3].

The accuracy of measurement and range of application, that can be attained with a combination of beam balance and magnetic coupling, mainly depend on three features of the coupling system:
1. the dead weight of the suspension
2. the dead capacitance
3. the conditions, that may be applied to the sample: atmospheric pressure, temperature and kind of atmosphere.

In the interest of accuracy and versatility, combination with high resolving commercial microbalances should be possible. On the other hand, the load capacitance should be sufficient to utilize the full range of semi-microbalances at least. The pressure, that may be applied to the sample, depends on the sturdiness of the separating wall. For higher pressures, this wall must be made of non-magnetic metal.

With coupling systems, which are based on the principle shown in Fig. 1, the accuracy of measurement as well as application in thermoanalytic systems is restricted by several disadvantages. First, the dead weight of the coupling is higher because of the control winding. Therefore, a balance with a high load capacity is required. Second, the power dissipation of the coil may cause convection within the casing of the balance. Third, wiring to the suspension is needed. These reasons make the coupling less suitable for microbalances.

Application in thermoanalytic systems is restricted because of the employed inductive or opto-electronical position sensors. These require a translucent or dielectric, that is, non-magnetic separating wall; otherwise, the sensor has to be placed inside the reaction chamber and only low pressures can be applied.

As only one degree of freedom is restricted, the floating magnet may twist around its axis. Therefore, the furnace to heat the sample has to be open at the top. A stable temperature-gradient is not possible, and disturbance because of convection can be anticipated.

Magnetic coupling for microbalances

Provided for the use in industrial and scientific research institutions, a new versatile magnetic coupling system allows gravimetric analyses of reaction systems as well as sorption analyses with an accuracy of ±1 microgram. The coupling was designed especially for microbalances, where low ballast weight is most important. No dielectrical materials are needed to separate the floating magnets from the balance. Besides, all electrical components of the coupling are installed outside the reaction chamber. This enables a simple and reliable construction, which can stand highest pressure as well as aggressive media.

As shown before, the coupling is inserted in the load suspension of the balance. According to the scheme, shown in Fig. 3, it consists of four bar

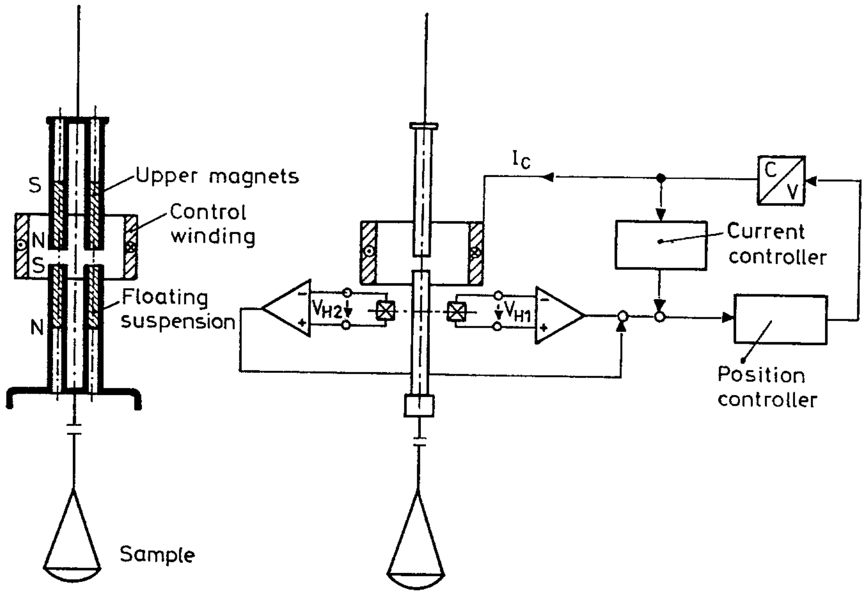

Fig. 3 Magnetic coupling for microbalances with extremely low coupling mass

shaped permanent magnets with axial polarization and a concentric coil to control the virtual mutual attraction by superimposing a magnetic field. Dead weight is reduced hereby, since the coil is mounted to the casing. Two pairs of magnets are used to prevent the floating part of the suspension from twisting around its axis. The position of the floating magnets is detected from outside by a magnetic sensor, consisting of two Hall-probes. This construction allows to use a metallic diaphragm. The Hall voltages depend on the radial component of the magnetic flux (see Fig. 4). The magnetic flux changes as well with the altitude of the magnets relative to the probe as with the respective horizontal distance. This means, that in the floating state swinging of the suspension will cause simultaneous vertical displacements. To avoid this, two Hall-probes are employed and a signal representing the vertical position only is formed by addition of the single voltages.

Unfortunately, the control winding exerts forces on the magnets, which do not always cancel. The disturbing difference of these forces depends on the degree of symmetry in the system and, certainly, on the controlling current. In

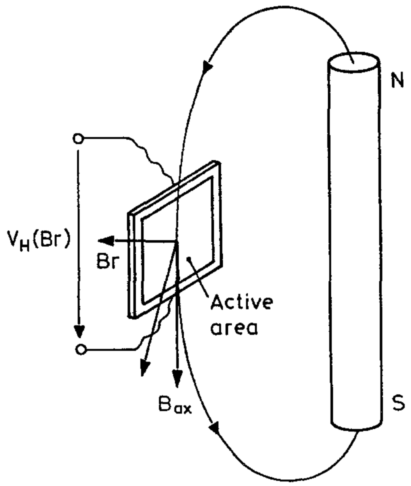

Fig. 4 Magnetostatic position detector Br/B_{ax}: radial/axial component of magnetic flux lines

order to keep this current minimal, a second control circuit, consisting of an integrator, is established. If a current flows through the control winding, the corresponding voltage drop across the winding causes the integrator to send a rising or falling voltage to the summing point of the position control circuit. Consequently, the position of the lower magnets will adjust to a distance, where the force required to keep the balance pan suspended is generated solely by the magnets. Only transient control currents are required to keep the system in a stable equilibrium. To avoid instabilities by interaction of the two circuits, integration time must be chosen large against the time constants of the position control circuit.

Magnetic system

The load capacitance of the system depends on the flux density of the applied magnets as well as on the thickness of the separating diaphragm. This thickness is the determining parameter for the range of pressure, which may be applied within the sample cell. With a given load capasitance and a minimum pole distance, the flux density should be not higher than required to keep the floating parts suspended. Thus, the volume and dead weight of the magnets as well as the stray flux are minimized. The magnets, however, must be strong enough to provide a sufficient flux density in the place of the Hall-probes in order to generate a reliable position signal. Using two pairs of rare-earth magnets with a diameter of 2 mm, a load capacitance of 4 gramm has been obtained with a tare of only 1.5 gramm. The minimum pole-distance is 3 mm. Due to the small diameter of these magnets, an enclosure can be made which stands several hundred bars.

Control technique

In principle, stable magnetic suspension can be realized with a PD-controller. When combined with an electromagnetically compensated bean balance, the control quality may be reduced because of interdependence between the two circuits. Thsi can be avoided by proper choice of the time constants of the position control circuit.

As shown before, the position of the floating magnets varies with the applied mass due to the auxiliary current control circuit. The non-linear relation between pole distance and attracting force causes a superproportional enhancement of open-loop-gain with increasing mass, and therefore the natural frequency of the closed loop changes. If there is a large variation in load, adaptive control may be necessary in order to keep the dynamic properties of the coupling sufficiently constant. The amplification of the position controller is increased with the applied load. Thus, high quality control, which is essential for measurement in the microgram range, can be maintained over a wide range of load independently from the dynamic properties of the balance.

Application in a thermogravimetric system for high pressured atmospheres

By thermogravimetric experiments between 1 and 10E(−5) bars many chemical reactions could be examined. On the other hand, thermal analysis at high pressures was rarely carried out. At present, thermal analysis is limited to some hundred bars. A thermogravimetric system for pressures up to 5,000 bars, which is suitable for aggressive media also, was realized by application of a modified version of the described coupling. This coupling is shown in Fig. 5.

To detect the vertical position of the floating magnets, two auxiliary flat magnets are employed additionally. These magnets are installed in the shaft of the floating suspension and polarized athwart the axis, in order to permeate thick separating walls. Using Hall probes with sufficiently large active areas, the linearity of the position signal is improved by averaging the flux density across an extended section.

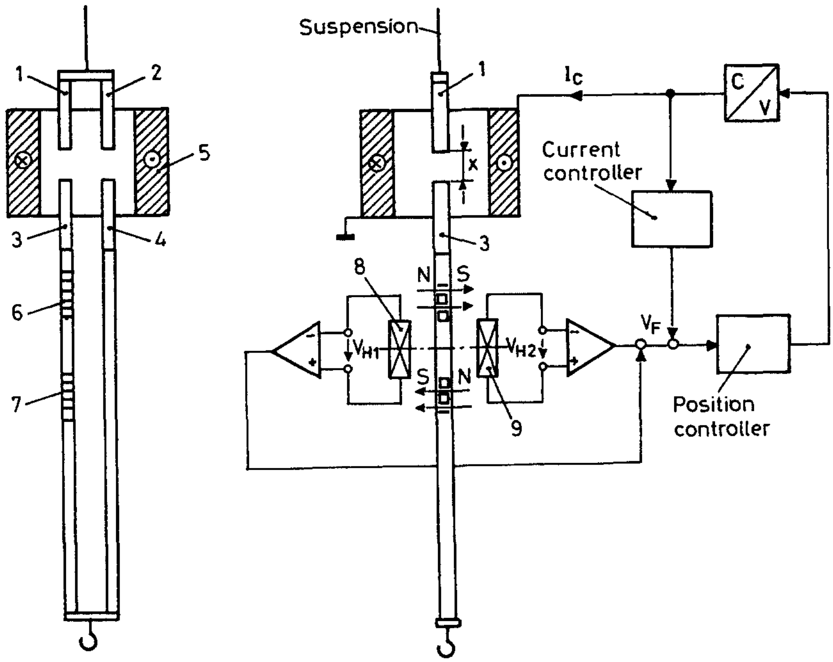

Fig. 5 Magnetic coupling for high pressure application; 1, 2 upper magnets; 3, 4 floating magnets; 5 control winding; 6, 7 auxiliary magnets; 8, 9 Hall probes

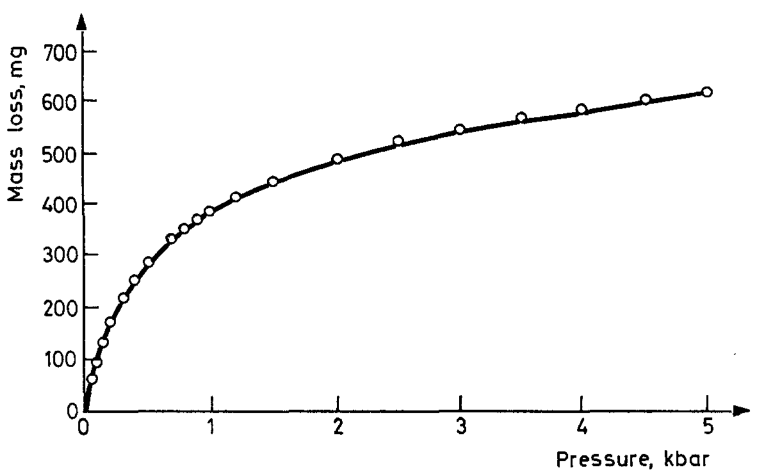

Fig. 6 Apparent loss of weight corresponding to a bouyancy effect (Weight of the floating magnet+pan : 1.5 g)

The system was tested over the full range of pressure. The apparent loss of weight of the pending magnets, corresponding to the bouyancy, is shown in Fig. 6.

References

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2 E. Robens, Th. Gast, E. Hoinkis and U. Müller, Fres. Z. Anal. Chem., 333 (1989) 428.
3 Th. Gast and G. Luce, A directly weighing suspension balance with frequency variant output, Ed. H. Wieringa, Mechanical problems in measuring force and mass, M. Nijhoff, Dordrecht 1986, p. 49.

Zusammenfassung — Mikrowaagen mit einer freien magnetischen Aufhängung ermöglichen eine ideale stoffliche Abtrennung zwischen dem Probenbereich und dem Waagenbereich. Dadurch werden Messbedingungen zugänglich wie Arbeiten unter extremen Drücken und unter spezieller, insbesondere aggressiver Atmosphäre, die besonders in der Thermogravimetrie von grosser Bedeutung sind.

Verschiedene Prinzipien der freien Aufhängung werden beschrieben und als Anwendungsbeispiel das Phänomen des Probenauftriebes in Abhängigkeit des Messdruckes dargestellt.