APPARATUS FOR MAGNETIZATION MEASUREMENTS IN GASEOUS ATMOSPHERE UNDER PRESSURE

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Руслан Бездушний, Радостина Дамянова. АПАРАТУРА ЗА МАГНИТНИ ИЗМЕРВАНИЯ В ГАЗОВА АТМОСФЕРА ПОД НАЛЯГАНЕ

Разработена е автоматизирана апаратура за измерване на намагнитеността при температури от 4,2 до 700 K в магнитни полета до 12 kOe във водородна атмосфера при налягания до 15 atm. Лабораторната постановка позволява изследване на температурната и полевата зависимост на намагнитеността на образците с контролирана концентрация на водорода в тях.

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An automated experimental apparatus for magnetization measurements in the temperature range from 4.2 to 700 K in magnetic fields up to 12 kOe under hydrogen atmosphere at pressures up to 15 atm has been constructed. This laboratory set-up allows studying the temperature and field dependencies of magnetization of samples with different hydrogen concentrations.

Keywords: vibrating magnetometer, intermetallic compounds, magnetization, hydrogen
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1. INTRODUCTION

The investigation of magnetic properties of hydrides (i. e. metal compounds containing hydrogen) is of great scientific and technological interest. However, the practical realization of such investigations is connected with considerable experimental difficulties. First, a great part of the hydrides are not stable compounds. The hydrogen quantity absorbed by them depends strongly on the temperature and pressure. Therefore, the standard methods for magnetic properties measurements are not applicable to them.

The second point is that the creating of new experimental equipment which allows one to measure the magnetic properties of samples as well as to control and keep their stoichiometric composition unchanged during the measuring process, is a complex scientific problem.

That is why, according to the available literature data, there are only few successful attempts to solve this problem. Frieske et al [1] describe an apparatus for measuring of magnetic susceptibility as a function of the hydrogen concentration (Faraday-type magnetometer). Yamaguchi et al [2] describe the construction of a vibrating sample magnetometer for magnetization measurements under hydrogen atmosphere (Foner-type magnetometer). There exists also another experimental apparatus for studying in situ the change of magnetic properties of intermetallic compounds upon hydrogen absorption [3, 4].

The present laboratory set-up allows carrying out magnetization measurements in situ in hydrogen (nitrogen and other gasses) atmosphere with controlled pressure. The usage of a personal computer as a part of the experimental apparatus facilitates and considerably accelerates the measuring process and realizes its partial or complete automation.

2. DESCRIPTION OF THE APPARATUS

The laboratory set-up was constructed on the principle of operation of a vibrating sample magnetometer and allows carrying out magnetization measurements in the temperature range from 4.2–700 K in magnetic fields up to 12 kOe and at hydrogen pressures up to 15 atm. It gives also the possibility to regulate the pressure and to control the hydrogen concentration in samples.

The block-diagram of this apparatus is shown in fig. 1.

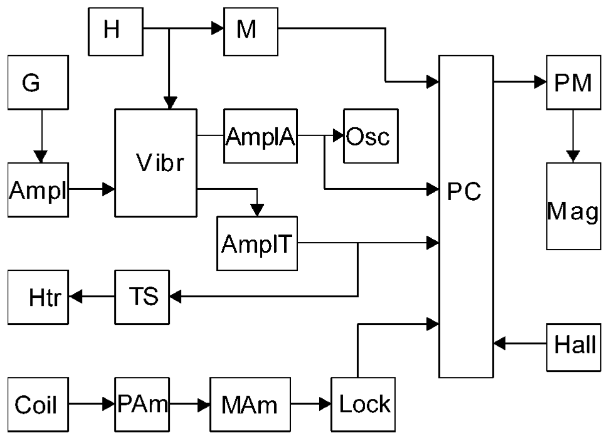

Fig. 1. Block-diagram of the laboratory set-up.
Mag – electromagnet, PM – power distribution system, PC – personal computer (registering system), Hall – Hall sensor, AmplT – thermocouple amplifier, TS – main thermostabilization system, Htr – heater, G – low-frequency generator, Ampl – low-frequency amplifier, Vibr – electromagnetic vibrator, AmplA – electromotive force amplifier, Osc – oscilloscope, Lock – synchronous detector, Coil – four balanced coils, PAm – coils signal preamplifier, MAm – measuring amplifier, H – pressure system, M – manometer with an electronic pressure sensor.

The apparatus comprises the following main systems:
– System of magnetic field creation;
– Temperature measuring and thermostabilization system;
– Mechanical system providing sample's vibration;
– Magnetization measuring system;
– Pressure system;
– Power system;
– Registering system.

The system for magnetic field creation provides a regulation of the magnetic field, which acts on the sample. This field is created by an electromagnet (Mag), which is fed by the electric power distribution system (PM). The regulation of the current through the magnet can be done either manually or automatically (PC). The value of the applied field is measured using Hall sensor (Hall).

Thermostabilization system is intended to keep up the temperature with an accuracy of 0.1 K. This system includes the copper-constantan thermocouple (situated in a high-pressure chamber near to the investigated sample), a measuring amplifier (AmplT), a main thermostabilization system (TS) and heater (Htr).

A low temperature chamber is used to carry out the magnetization measurements in the temperature range up to 300 K. The sample's cooling is realized by pumping off through the low temperature chamber liquid nitrogen (helium) or its vapour.

The high temperature chamber is for carrying out measurements in the temperature range from 300 to 700 K.

During the measuring process the sample is either in controlled gaseous atmosphere or under vacuum.

The mechanical system includes a low-frequency generator (G), an amplifier (Ampl) and an electromagnetic vibrator (Vibr). The sample is placed in a special holder situated in a high-pressure chamber at the end of the mount.

The mount's motion is controlled by the help of an electromagnetic sensor (situated in the electromagnetic vibrator). The sensor is a hardly fixed to the mount measuring coil, which moves in permanent magnetic field. The electromotive force induced in it is proportional to the rate of mount's shifting. The electromotive force is amplified (AmplA) and after that goes to the oscilloscope (Osc), to the synchronous detector (Lock) and to the registering system (PC).

The magnetization measurements are carried out using a standard method (with the help of four balanced coils (Coil), situated between the pole-shoes of the magnet). The measuring system includes measuring coils, a preamplifier (PAm), situated between the magnet poles next to the measuring coils, a measuring amplifier (MAm), a synchronous detector (Lock) and a registering system (PC). The usage of the synchronous detector allows measuring not only the value, but also the direction of the magnetization vector.

The pressure system (H) is intended for keeping up the necessary (with given parameters) working gaseous atmosphere around the sample. The system includes:

– Balloon containing working gas under pressure up to 150 atm;
– Reducible valve, keeping up and regulating the pressure (from 0 to 25 atm);
– Backing pump, pumping off the pressure system;
– Electromagnetic valves system, controlling the working regime of the pressure system and the pressure measuring block.

A distinctive feature of the apparatus is the specific construction of the high pressure chamber (fig. 2). The hermetically sealed connection is moved away from the sample and its temperature is about the room temperature. Thus, it is possible to extend the temperature range towards the higher temperatures. The upper limit depends on the mechanical properties of the chamber material only.

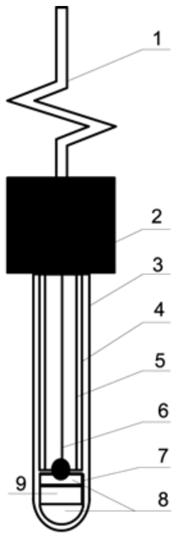

Fig. 2. Construction of the high pressure chamber:
1) Copper pipe, 2) Hermetically sealed connection, 3) Chamber, 4) Quartz tube, 5) Glass tube, 6) Thermocouple, 7) Quartz ampoule, 8) Quartz wool, 9) Powder sample.

A manometer with an electronic pressure sensor (M) connected with the registering system have been used to register the pressure.

The personal computer (PC) appears to be the main part of the registering system. A 16-channel analog-digital transformer has been used to transform the analog signals entering the registering system into digital form. A special program, controlling and ruling all the processes, has been worked out.

On the figure 3 the temperature dependencies of the magnetization of the $ErFe_{11}Ti$ and its hydride are shown. It is visible, that the hydrogen essentially raises the Curie temperature. That can be explained by the increase of the interatomic distances and the reduction of negative exchange interactions between the iron atoms. The created equipment has been used for the magnetic properties research of the hydrides of the intermetallic compounds $RFe_{11}Ti$, $R_2Fe_{14}B$ and others (for example, see [5, 6]).

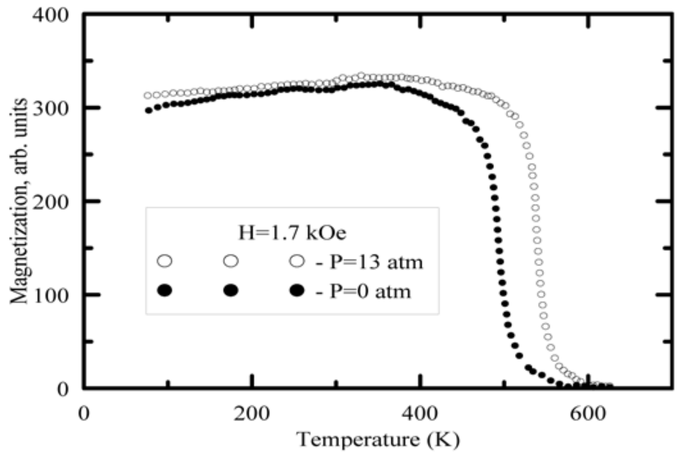

Fig. 3. Temperature dependencies of the magnetization of the intermetallic compound ErFe11Ti and its hydride.

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