Z23 - Resistometric Studies of Magnetic and Order-Disorder Structural Phase Transitions in Intermetallic Compounds

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Resistometry is a convenient and effective tool for detection of phase transitions in bulk conductors. The aim of this lab is to observe and analyze anomalies in the temperature dependence of resistivity of FePd intermetallic compound which are associated with atomic ordering process and changes in magnetic ordering of the sample during heating and cooling.

Preparatory questions

1. Electrical conductivity of metals as derived from free electron gas model (electrical conductivity, Ohm's law, temperature dependence of electrical resistivity, lattice, residual and magnetic contributions to the electrical resistivity [1, 2, 3].
2. Resistivity determination methods (including a four probe method)[1, 2].
3. Temperature measurements by a termocouple with electronic compensation [1].
4. Phase transitions and their classification [1, 2].
5. Magnetic phase transitions (e.g. from ferromagnetic to paramagnetic state) [1, 2].
6. Crystals, crystal structure, defects in crystals [1, 2].
7. Atomic migration in alloys. Activation energy. Order-disorder phase transition [1, 2].
8. Construction and principle of operation of a rotary vane pump, a pressure reducer, a standard reference resistor and a thermocouple [4].

Computational assignments

Temperature coefficient of resistivity for copper is reported to be equal to $3.9\text{x}10^{-3}\text{K}^{-1}$. Calculate the maximal allowed change in temperature if the relative change in resistivity should not exceed 2%.

Apparatus and materials

The intermetallic FePd compound, that undergoes magnetic and structural phase transitions, is investigated (the phase diagram is available from [1]). The experiment is carried out in an inert gas atmosphere (Ar) in the temperature range between room temperature and about 900K. Prior to the lab, the sample is mounted in the apparatus and annealed at the temperature slightly below the temperature of the structural phase transition and then slowly cooled down to room temperature.

Measurements of resistivity (by four-probe method) versus temperature (measured by means of a thermocouple with electronic compensation) and data acquisition is computer controlled (program written in LabView). The functional drawing of the experimental setup is shown in Fig.1.

The aim of the student is to prepare the experimental setup for the measurements, program the temperature controller to assure that the sample will be heated/cooled with a constant rate and control the data collection during heating and cooling - all in close collaboration with the experiment supervisor.

Experiment

1. Prepare the detailed program of the experiment and discuss it with the experiment supervisor. Remember to set the same range for the changes of the temperature in the regions of interest (i.e. the regions you expect the phase transitions) both during heating and cooling.
2. At the presence of the experiment supervisor switch on the temperature controller, the multimeter, the computer and run the data acquisition program (written in LabView). Then program the temperature controller according to the experimental plan prepared in the point 1.
3. Check the pressure at the output of the pressure controller connected to the gas cylinder filled with Ar. There should be a small overpressure (about 0.2 bar). Using the appropriate valves flush three times the sample chamber with Ar by sequential pumping followed by refilling. Leave the sample chamber connected to the Ar gas cylinder and till the end of the experiment keep the low (about 0.2 bar) overpressure (explain why it is recommended). Switch off the pomp (remember to open the pump venting valve).
4. If the heater is in the upper position move it down to cover the quartz sample chamber.

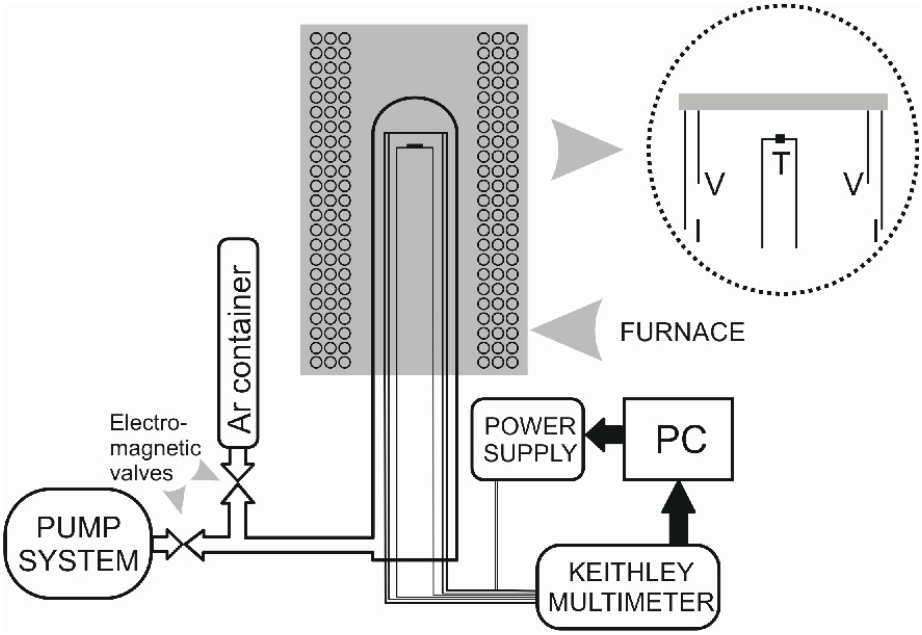

Figure 1: Functional drawing of the experimental setup for resistomeric measurements above room temperature. The sample is placed inside the quartz sample chamber (filled with Ar). The inset in the upper right corner shows the sample with voltage and current contacts (for resistivity measurements by four-probe method) and a thermocouple that measures the temperature of the sample. Another thermocouple is placed inside the furnace and connected to a temperature controller (not shown in the drawing).

5. Run the temperature controller program and the experiment in the data aquisition program. Open the water valves, switch on the water flow and the heater on the control panel.

6. Complete data acquisition during heating and cooling in the chosen temperature range. Keep checking the pressure in the quartz experimental chamber not allowing to grow higher than 0.4 bar (if necessary switch on the pump, unpump the experimental chamber slightly and fill it with Ar).

7. After completing the data during heating, copy the data to a new file and start their analysis.

8. After completing the whole experiment switch off the heater and the data acquisition program. Copy the data to your files. Close the water valves, the connection to the Ar cylinder and switch off the control panel.

During the experiment keep writing detailed notes *in the apparatus logbook* in the way making possible to reproduce each step of your work and values of all parameters of the experiment. After completing the experiment copy the entry to your lab book.

Data analysis

1. Determine the Curie temperature and the temperature of the order-disorder phase transition. It could be done e.g. in Origin (available on the computers in the lab) by finding the minimum of the second and first derrivative of R, respectively.

2. Estimate uncertainties of the determined phase transitions. You should take into account not only those coming from calculation procedures you have used but try also to estimate the influence of the experimental procedure and the experimental setup you have used.

3. Discuss the kinetics of the observed phase transitions basing on the analysis of the temperature dependence of resistivity, registered in the experiment during heating and subsequent cooling.

References

[1] Lab-manual available from (you have to log in):.

[2] Handbooks on solid state physics (e.g. C. Kittel, *Introduction to Solid State Physics*, John Wiley & Sons 1996).

[3] http://hyperphysics.phy-astr.gsu.edu/.

[4] Handbooks on experimental physics and information available in the Internet.