Z21 – Martensitic Transformation in Shape Memory Alloys Investigated by Resistometric Method

Physics Laboratory II

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The aim of this laboratory class is to introduce resistometry as an important and convenient experimental technique used to study structural phase transformations in conducting crystalline solids. This aim is realized by observation of resistivity hysteresis loop in the Ni-Ti alloy which has vast medical (e.g. facial-jaw surgery and braces) and industrial applications.

The martensitic transformation strongly influences electrical resistivity thus this kind of transformation can be easily investigated by measuring electrical resistivity vs. temperature dependence.

Preparatory questions

1. Phase – definition, classification [1].
2. Martensitic transformation [2]
3. Theory of electrical conductivity of metals as derived from the free electron Fermi gas model (electrical conductivity, Ohm's law, temperature dependence of electrical resistivity, lattice and residual contributions to the electrical resistivity) [3].
4. Construction and principle of operation of rotary vane pump, standard reference resistor, thermocouple and pressure reducer [4].

Note
The Ni-Ti alloy which is investigated in this laboratory class, commonly known as **nitinol**, is a shape memory alloy and has vast medical (surgery, braces) and industrial applications. Those especially interested in possible applications can find a lot of information in the Internet.

Computational assignments

1. Temperature coefficient of resistivity for copper is reported to be $\alpha_{Cu} = 3.9 \cdot 10^{-3}\ \mathrm{K}^{-1}$. Calculate the relative change in electrical resistance of copper wire related to temperature rise from 300 up to 330 K.
2. Resistance of the sample was determined by driving a current of 50 mA. Calculate the corresponding voltage drop assuming that the sample resistance is equal to 2 mΩ.

Apparatus and materials

The experimental apparatus is presented in figure 1 and it includes the following equipment and elements:

- computer controlled data acquisition system for measuring electrical resistivity by four-contact method
- open-flow nitrogen cryostat
- cryogenic equipment
- rotary vane pump

Liquid nitrogen present in a cryogenic storage dewar evaporates producing a cold nitrogen vapor. The cooling effect is obtained by vaper flow through the cryostat. The experimentalist can control both the direction as well as the rate of sample temperature changes.

Experiment

1. Preliminary actions:
   (a) Switch on the rack, computer, Z_3 power supply (see fig. 1), voltmeters and rotary vane pump (if the pump is equipped with a venting valve, please, ensure that the valve is closed).
   (b) Start the data acquisition program.

   During this time the laboratory staff is mounting a cryogenic storage dewar filled with liquid nitrogen.

2. Cryostat Preparation:
   (a) Check the pressure at the output of pressure controller which is connected to the gas cylinder filled with helium. There should be a small overpressure (~0.2 bar). Using the K_2 ball valve flush twice the sample chamber with helium by sequential pumping followed by refilling. Leave the sample chamber connected to the helium gas cylinder as it would reduce pressure deviations induced by temperature changes.
   (b) If the cryostat is equipped with a vacuum insulation chamber, leave it connected to the pump during experiment. Otherwise swith

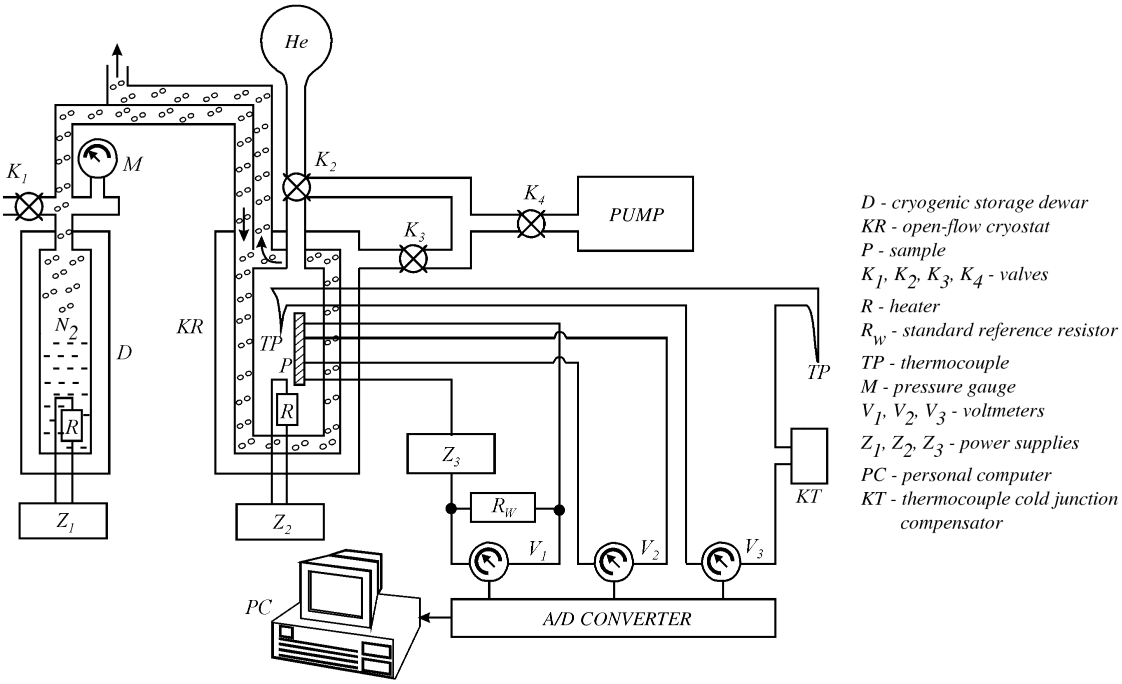

Figure 1: Experimental setup.

off the pump (remember to open venting valve if it is available).

3. Collecting sample resistance vs. temperature data while cooling and heating:

   (a) Start data acquisition.

   (b) Switch on the sample heater and heat the sample up to 40^{o}C.

   (c) **<u>Just</u> after reaching 40^{o}C switch off the sample heater.**

   (d) Switch on the heater installed in the dewar.

   (e) Cool down the sample to the lowest available temperature (approx. -130^{o}C: the lowest temperature depends on experimental set – please consult laboratory assistant responsible for your class). It is only possible if the nitrogen vapor pressure is high enough. **Your job is to control the vapor pressure and keep it just below the limit marked by red color on the pressure gauge. If the pressure starts to exceed the limit switch off the dewar heater. If it does not help release the safety valve located at the top of the dewar.**

   Note: Effective cooling is only possible with the pressure at the limit. Do not reduce the pressure without substantial need.

   (f) Just after reaching the lowest available temperature switch off the dewar heater and switch on the sample heater.

   (g) Heat the sample up to 40^{o}C trying to keep the heating rate equal to the cooling one. It can be obtained by controlling the sample heater current and gradually reducing nitrogen vapor pressure by operation of dewar safety valve. Please consult laboratory assistant at which temperature the safety valve can be left open.

   (h) **<u>Just</u> after reaching 40^{o}C swith off the sample heater.**

   (i) **Remember to leave the dewar safety valve open** as it would prevent nitrogen vapor pressure from exceeding the limit.

   (j) Copy your experimental data and switch off the pump (remember to open venting valve if it is available), computer, voltmeters, Z_3 power supply and rack.

Data analysis

Detailed analysis of processes occuring in the Ni-Ti alloy leads to the following conclusions:

- Above room temperature a phase of the bcc-type structure ($a = b = c,\ \alpha = \beta = \gamma = 90^o$) is stable.

- With decrease of temperature an orthorhombic phase ($a \neq b \neq c,\ \alpha = \beta = \gamma = 90^o$) starts to develop at T_R (this phase is denoted in fig. 2 as R). The appearance of the R phase precedes martensitic transformation and is connected with increase of resistance.

- In case of the investigated Ni-Ti alloy its martensitic phase has rhombohedral structure ($a = b = c,\ \alpha = \beta = \gamma \neq 90^o$). The growth of martensitic phase starts at temperature M_s and manifests

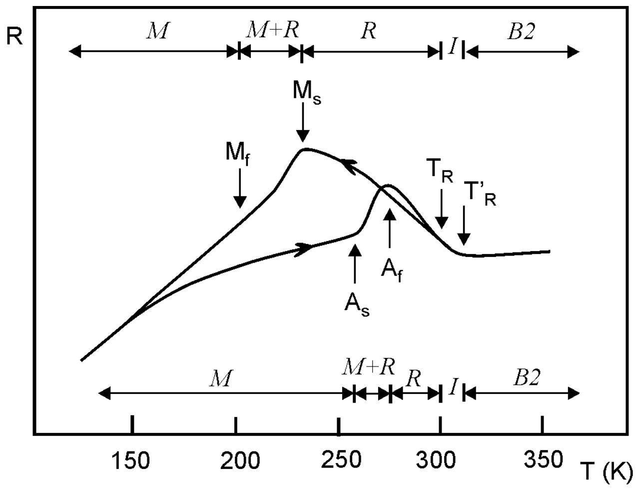

Figure 2: **Illustrative** plot of resistance vs. temperature dependence for nitinol sample undergoing a martensitic transformation. Please, notice the temperature ranges for particular phases and characteristic temperatures.

itself in decrease of resistance. The martensitic transformation in nitinol is a two-step process as visible in the shape of resistance vs. temperature curve shown in fig. 2. The curve becomes linear at temperature M_f which is the final temperature of martensitic transformation.

- While heating martensitic phase a retransformation (called also a reverse transformation) starts at temperature A_s at which resistance vs. temperature curve becomes non-linear. The disappearance of martensitic phase is associated with growth of the orthorhombic phase R and manifests itself in increase of resistance until A_f temperature (final temperature of retransformation process) is reached.

The obtained resistance vs. temperature can be analyzed in the following way:

- Determination of the M_s and A_f temperatures: find temperatures corresponding to the main maxima in the resistance vs. temperature curves recorded while cooling and heating, respectively. It can be done, for example, by determining the temperature at which the derivative changes its sign.

- Determination of the M_f and A_s temperatures: fit linear function below investigated temperatures, i.e. beyond temperature range where (re)transformation takes place. Using the fit determine temperatures where the resistance vs. temperature curve becomes non-linear.

- Estimate uncertainties of determined chracteristic temperatures.

Safety rules

- **Mounting the cryogenic storage dewar is done by laboratory staff only. Please, remember that direct contact with liquid nitrogen leads to frostbites!**

- Do not exceed the nitrogen vapor pressure limit marked by red color on the pressure gauge.

- After finishing your measurements leave the dewar safety valve open.

References

[1] Handbook on thermodynamics (e.g. D. Elwell, A.J. Pointon, *Classical thermodynamics*, Penguin Books Ltd 1972).

[2] M. Ahlers, *Revista Matéria* 9(3), pp. 169-183 (2004) (http://www.materia.coppe.ufrj.br/sarra/artigos/artigo10308).

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

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