D2. Anelasticity of Solids

I. OBJECTIVE OF THE EXPERIMENT
We will measure the elastic and anelastic modules of different metals, observe the relaxation phenomenon in steel as well as Snoek relaxation.

II. PHENOMENOLOGY OF ANELASTICITY
When a solid that can’t move is subject to a set of external stresses, it reacts by deforming. For small stresses, the deformation is instantaneous, proportional to the applied stress, and vanishes when the stress is removed. This is called elastic deformation. The relationship between stress \( \sigma \) and strain \( \varepsilon \) is given by Hooke’s law:

\[
\sigma = E\varepsilon \quad \text{where } E = \text{Young modulus}
\]

or

\[
\varepsilon = J\sigma \quad \text{where } J = \text{elastic complaisance}
\]

Anelasticity in solids can be shown by this simple experiment (fig.1a). A stress \( \sigma \), of low intensity is applied to a sample at the time \( t = 0 \), and is held constant, while the strain \( \varepsilon \), is recorded. We observe an instantaneous strain, \( \varepsilon_e = J_u\sigma \), where \( J_u \) is called the unrelaxed complaisance, and a so-called anelastic strain, \( \varepsilon_a \), that increases over time from zero to a limiting value, \( \varepsilon_a^\infty \). When in equilibrium:

\[
\varepsilon = \varepsilon_e + \varepsilon_a^\infty = J_r\sigma
\]

where \( J_r \) is the relaxed complaisance

Fig. 1: Anelasticity of solids

The process through which a solid under a stress \( \sigma \) goes from one equilibrium state at \( t = 0 \), to another one at \( t = \infty \), is called anelastic relaxation, and is defined by two main parameters.

- relaxation intensity \( \Delta = \frac{\varepsilon_a^\infty}{\varepsilon_e} = \frac{J_r - J_u}{J_u} \) et

- relaxation time \( \tau \)

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\( \tau \)
If after a certain amount of time, the stress is removed, we will see the elastic strain vanish instantly, and the anelastic strain decrease with a relaxation time $\tau$ (fig 1). In this experiment, the deformation is completely reversible. The difference between the elastic and anelastic process, is that the latter isn’t instantaneous. In metals, anelastic strain is much smaller than elastic strain, i.e. $(J_r - J_u) \ll J_u$.

We can describe the evolution of the anelastic strain $\varepsilon_a$ with an exponential:

$$\varepsilon_a = \varepsilon_a^\infty (1 - e^{-t/\tau})$$  \hspace{1cm} (2)

a) Elastic strain $\varepsilon_e$ and anelastic strain $\varepsilon_a$ of a solid under a stress $\sigma$.

b) Rheological model corresponding to the anelastic process in fig. 1.

From equation (2) and in the case where $(J_r - J_u) \ll J_u$, we can determine the equation of an ideal linear solid, characterizing the process described in fig. 1

$$\varepsilon + \tau \cdot \dot{\varepsilon} = J_r \cdot \sigma + \tau \cdot J_u \cdot \dot{\sigma}$$  \hspace{1cm} (3)

From a rheological standpoint, the ideal linear solid can be represented by the model in figure 1b (to be shown by the student)

The measurements of the system in a static system are very delicate, since the average anelastic strain in metals is generally of the order of $10^{-9}$ to $10^{-8}$, which is why we use dynamic methods to determine anelastic strain.

A harmonic stress of frequency $\omega$, $\sigma = \sigma_0 \exp(i \omega t)$, is applied to the solid. The linearity of “stress-strain” relationships results in a strain $\varepsilon$ that is also periodic, and of same frequency $\omega$, $\varepsilon = \varepsilon_0 \exp(i(\omega t - \delta))$, however, it will be phase-shifted with respect to the strain by an angle $\delta$ (due to anelasticity).

Introducing the expressions of $\sigma$ and $\varepsilon$ in equation (3), yields:

$$\varepsilon = J^\ast(\omega)\sigma = (J_1(\omega) - iJ_2(\omega))\sigma$$  \hspace{1cm} (4)

where $J_1$ and $J_2$ are respectively the real and imaginary parts of the complex complaisance $J^\ast$.

$$J_1 = \left(\frac{J_u + (J_r - J_u)}{1 + \omega^2 \tau^2}\right),$$

$$J_2 = \frac{(J_r - J_u) \cdot \omega \tau}{1 + \omega^2 \tau^2}. \hspace{1cm} (5)$$

Measuring the phase-shift between stress and strain, or measuring the tangent of the mechanical loss angle ($\tan \delta$), is actually a measure of the internal friction of the material $Q^{-1}$

$$Q^{-1} = \tan \delta = \frac{J_2}{J_1}$$  \hspace{1cm} (6)

By introducing (5) in (6) and supposing that $(J_r - J_u) \ll J_u$, we get:

$$Q^{-1} = \Delta \frac{\omega \tau}{1 + \omega^2 \tau^2} \hspace{1cm} \text{et} \hspace{1cm} \frac{\delta J}{J} = \frac{J_1(\omega) - J_u}{J_u} = \Delta \cdot \frac{1}{1 + \omega^2 \tau^2} \hspace{1cm} (7)$$

where $\frac{\delta J}{J}$ is the variation of the complaisance due to anelastic relaxation.
Equations (7) show that the internal friction, $Q^{-1}$, has a maximum that depends on $\omega \tau$, centered in $\omega \tau = 1$. This maximum, or internal friction peak gives us the intensity $\Delta$ of the relaxation, as well as the relaxation time $\tau$, (position of the peak on $\omega \tau$).

![Diagram](image)

Fig. 2: Internal friction peak (Debye peak) and default associated elastic modulus.

From a microscopic point of view, anelastic deformation can be interpreted as the motion of defects of the crystalline structure (elastic dipole associated to point defects, dislocations, grain boundaries, ...) that go from one equilibrium state at $\sigma = 0$ to a new equilibrium state for $\sigma$. The intensity of the relaxation $\Delta$, is proportional to the defect concentration, and the relaxation time $\tau$, is a measure of their mobility. When the mechanisms that control the mobility of the defects are thermally engaged, we have:

$$\tau = \tau_0 e^{\frac{H}{kT}} \quad (H = \text{activation enthalpy})$$

And for an imposed frequency $\omega$, we can see a peak that depends on the temperature $T = T_p$ such that:

$$\omega \tau = \omega \tau_0 e^{\frac{H}{kT}} = 1$$

from (7) and (8):

$$Q^{-1} = \Delta \frac{1}{2ch \frac{H}{k} \left( \frac{1}{T_p} - \frac{1}{T} \right)}$$

The height of the peak is $\frac{\Delta}{2}$ (fig 2).

**Measuring the activation enthalpy $H$**:

1st method: Perform measurements at different frequencies. For the frequency $\omega_i$, the peak is located in $T_{p_i}$. We then plot $\ln \omega_i$ versus $\frac{1}{T_{p_i}}$, whose slope is $(-H/k)$.

2nd method: From equation(9) we know the value of $Q^{-1}$ at mid-height (i.e. at a temperature $T_i$)

$$Q^{-1} = \frac{\Delta}{4} = \Delta \frac{1}{2ch \frac{H}{k} \left( \frac{1}{T_p} - \frac{1}{T_i} \right)}$$

Comparing (9) and (10) yields:

$$ch \frac{H}{k} \left( \frac{1}{T_p} - \frac{1}{T_i} \right) = 2$$

thus

$$\frac{H}{k} \left( \frac{1}{T_p} - \frac{1}{T_i} \right) = 1.317$$

or

$$H = \frac{2.633k}{\Delta \left( \frac{1}{T} \right)} \quad \text{with} \quad \Delta \left( \frac{1}{T} \right) = \frac{1}{T_2} - \frac{1}{T_1} \quad \text{width of peak mid-height}$$

(11)
Anelastic Snoek relaxation

Snoek relaxation is due to interstitial point defects in the form of solid solutions of C, O, N, H, in centered cubic (c.c.) crystals, such as carbon of iron.

These impurities are in the octahedral sites of the c.c. structure. The generate elastic dipoles, of tetragonal symmetry. The axis is parallel to the 4-symmetry axis of an octahedron.

For instance in figure 3, the interstitial atom located in position 1 can create a dipole of axis parallel to Ox1. It will push back the neighboring atoms (up and down in the figure).

Interstitial atoms can be divided into three categories, since the axis of the dipole can be oriented according to Ox1, Ox2 or Ox3.

Let \( N_0 \) be the total number of defects or elastic dipole per unit volume. Then:

\[
N_0 = N_1 + N_2 + N_3 = \sum_{i=1}^{3} N_i
\]

(12)

where \( N_i \) is the concentration of dipoles whose axis is parallel to \( x_i \).

When in equilibrium, and for no external stress (\( \sigma = 0 \)), the 1,2,3 sites are equivalent.

Then:

\[
N_1 = N_2 = N_3 = \frac{N_0}{3}
\]

(13)

and the extra elastic deformation due to the interstitial defects is the same in all three directions. When we apply stress in a given direction, one type of site can be preferred, and the defect densities will vary.

\[
N_1 \neq N_2 \neq N_3 \neq \frac{N_0}{3}
\]

(14)

In order to simplify, consider a stress parallel to Ox1 (\( \sigma = \sigma_{11} \)) and observe the corresponding strain \( \varepsilon = \varepsilon_{11} \). We have:

\[
\varepsilon = \varepsilon_\sigma + \varepsilon_a = J_n \sigma + \Delta \lambda (C_1 - \frac{c_0}{3})
\]

(15)

where

\[
\Delta \lambda = \left. \frac{\partial \varepsilon}{\partial C_1} \right|_{c_0 = \text{const}}
\]

(16)

\( \Delta \lambda \) defines the strain variation due to migration of defects from 2 and 3 towards 1. \( c_0 \) is the atomic concentration of interstitials, \( c_1 \) represents those oriented along Ox1. The anelastic deformation is due to the extra strain generated by this atomic movement.

\[
\varepsilon_a = \Delta \lambda (c_1 - \frac{c_0}{3})
\]

(17)
We can calculate the relaxation parameters $\Delta$ and $\tau$:

$$\Delta = \frac{2}{9} \frac{c_0 v_0}{J_p k_b T_p} \Delta \lambda^2 \quad \text{and} \quad \tau = \frac{a^2}{36D}$$

(18)

where $c_0$ is the atomic concentration of interstitials, $v_0$ their atomic volume, $D$ their diffusion coefficient, and $a$ the lattice parameter:

$$D = D_0 e^{-\frac{H}{k_b T}}$$

(19)

where $H = $ diffusion energy (enthalpy) of the interstitials, $k_b$ the Boltzmann constant. The relaxation intensity is proportional to the concentration of relaxing atomic interstitials. The relaxation time is a measure of the activation energy of their diffusion.

### III. EXPERIMENTAL PROCEDURE

tg$\delta$ can be obtained directly by measuring the phase-shift between stress and strain. However, for weak damping ($\tan \delta \sim 10^{-3}$), it can be easier to use a resonant system, vibrating at its eigenfrequency. In this experiment, we will use a short vibrating rod (fig. 4)

The vibration frequency "$f$" of an unattached rod is:

$$f = \frac{1.03}{l^2} \sqrt{\frac{E}{I^2 \rho}}$$

(20)

Where $E$ is the Young modulus, $\rho$ the density, $e$ and $I$ the thickness and $l$ the length of the sample. Inverting equation (20) yields $E$:

$$E = 0.943 \frac{Dl^4}{e^2 f^2}$$

(21)

Fig. 4: Flexion of a rod of length $l$. The vibration knots are located at 0.224 $l$ from the end of the rod.

The internal friction, $Q^{-1}$, is determined by the decrement rate of the oscillation amplitude (fig. 5)

$$\tan \delta = Q^{-1} = \frac{1}{n \pi} \ln \frac{A_i}{A_{i+n}}$$

(22)

where $A_i$ and $A_{i+n}$ are the $i^{th}$ and $(i+1)^{th}$ vibration amplitudes respectively (fig. 5)

Fig. 5: Free decrement of an oscillating system.
Measurement setup (fig. 6)

The sample to measure is a small rod of length 1 and thickness e that is hung from four thin steel wires that go through the knots of the fundamental vibration (cf. fig. 4). An electrode is placed in the middle of the sample (fig. 6), and thereby forms a capacitor of capacity C. When a voltage V between the electrode and the sample, and electrostatic force F is applied on the rod. For a harmonic voltage:

\[ V = V_0 \cos \frac{\omega t}{2}, \text{ the force is } F = \frac{CV_0^2}{4d}(1 + \cos \omega t) \quad \text{with} \quad C = \varepsilon \frac{S}{d} \quad (23) \]

where \( \omega = \) frequency, \( \varepsilon = \) permittivity of the medium, \( d = \) electrode-sample distance and \( S = \) electrode surface. The force F sets the metal rod into vibration. Since C is part of a system oscillating at high frequency (HF), the variation of C (i.e. variation d) generates a high frequency modulation v.

\[ v = \frac{1}{2\pi \sqrt{LC}} \quad \text{thus} \quad \frac{\Delta v}{v} = -\frac{\Delta C}{2C} \quad (24) \]
The modulated high frequency goes through an FM demodulator (same principle as FM radio), whose output is an electric voltage proportional to the capacity variation $C$, i.e. the vibration of the sample. This signal is filtered, amplified using a controlled gain amp (in order to get a constant oscillation amplitude), phase-shifted, and reinjected into the circuit using an power amplifier. The oscillations are being maintained using a closed circuit. The electric signal that exits the FM demodulator represents the sample’s oscillation. The frequency meter can measure the frequency $f$ of the electrical signal, which yields the elastic modulus $E$ (equation (21)). An electronic switch regularly cuts the excitation, so that we can observe the free decrement of the oscillations (fig. 5), which yields the internal friction according to (22). In our case...
(frequencies of the order of the kHz), the amplitudes $A_i$ and $A_{i+1}$ are determined by electronic thresholds, and the device counts the number of oscillations between these values. The device directly calculates the Q-factor ($Q = 1 / Q^{-1}$).

In an attempt to avoid friction with air and electric discharges between the electrode and the sample and oxidation of the heated sample, the whole device is placed in a vacuum. The vacuum is achieved with a standard system made of a rotary vane pump ($P_{atm} \rightarrow 0.01$ mbar) and a turbo-pump ($0.01$ mbar $\rightarrow 10^{-4}$ to $10^{-6}$ mbar), cf. experiment. K1, Vacuum technology.

The sample's temperature is regulated using a PID temperature regulator, made of:

1) A chromel-alumel thermocouple (K-type) placed on the sample holder. The thermocouple supplies a voltage that depends on the temperature difference between the sample and the measuring device.

2) A Eurotherm regulator, that compares the sample temperature $T_{ech}$ to a reference temperature $T_{ref}$. Depending the temperature difference $\Delta T = T_{ref} - T_{ech}$, it will heat the sample more or less, i.e. will output a voltage $V_s$ between 0 and 5 Volts. The PID response (Proportional, Integral, Derivative) is given by:

$$V_s = K(\Delta T + \frac{1}{t_i} \int_0^t \Delta T dt + \frac{1}{t_d} \frac{d\Delta T}{dt})$$

with $K =$ proportional gain, $t_i =$ integration time, $t_d =$ derivation time

3) A power amplifier that supplies the oven (resistive thermocoax of 25Ω), under the form of a continuous voltage 0 .. 100 V, 0 .. 4 A, as a function of the command voltage $V_s$ (0 V = 0 % power, 5 V = 100 % power).

IV. SUGGESTED EXPERIMENTS

1. Measure of the elastic modulus $E$ of a few materials (steel, copper, aluminum, magnesium) as well as their vibration absorption capacity $Q^{-1}$ at room temperature.

2. Study the Snoek relaxation due to carbon in a ferritic steel. On order to do so, measure the internal friction and frequency (elastic module) as a function of temperature, for values ranging from room temperature to approx. 250°C. Plot the graphs $Q^{-1}(T)$ and $E(T)$ and calculate the diffusion energy and the interstitial carbon concentration from the peak data (subtract the background!) (see equation (9) and (18)), using the following values: $\Delta \lambda = 0.62$, $k = 1.38 \cdot 10^{-23}$ J/K, $v_o = 3.05 \cdot 10^{-30}$ m$^3$.

a) Sample setup and measures.

With the valves V1, V2 and V3 close, open the setup by removing the long steel tube. If the chamber is in a vacuum (see bourdon gauge), let air in the system through the microvalve. Remove the oven, and move the electrode up using the micrometric screw. Then, place a sample between the wires, make sure it's well centered, and move the electrode back down, close to the sample (see fig. 8). Close the setup, with or without the oven, depending on the measurements you would like to gather. Perform a high vacuum (see point "c" below for detailed instructions)

when the vacuum exceeds 0.01 mbar, start the TECTANEL 1000 device (Fig. 7).
With the excitation amplitude on minimum, move the electrode closer using the micrometric screw, until getting a short circuit (short circuit light on TECTANEL 1000 panel). Move the electrode away by 0.1 mm. Then, look for the HF frequency using the Tuning button (needle centered on 0, AFC off). Set the excitation to the maximum, place the gain on ~100, and look for the vibration frequency of the sample using the coarse frequency and fine frequency buttons (1–10 kHz). Then, adjust the excitation amplitude in order for the needle to be between 1 and 1.5 Volts. Place the Measurement Rate on Rec, for the device to measure the frequency $F$, the temperature $T$ (not connected here), and the Q-factor $Q$.

b) Temperature variation

Verify the connection: thermocouple $\rightarrow$ Eurotherm $\rightarrow$ DC power supply (100 V, 4 A) $\rightarrow$ 25 Ω thermocoax. oven

Turn on the Eurotherm 912P (Fig. 9)
Simplified instructions for Eurotherm (Fig. 9):

**Button “6”:** short push: switch from "SP" to "OP" to "TIME"
- long push, enter prog. mode.—> PROG
Then press button "1" to see the 8 ramps levels
\[ Pr(i) = i^{th} \text{ ramping speed in } ^\circ C/\text{min} \]
\[ PI(i) = i^{th} \text{ level in } ^\circ C \]
\[ Pd(i) = \text{length of } i^{th} \text{ level in minutes} \]
Change the values using buttons "4" and "5"

**Button “2”:** Starts the program. A second hit puts the system on hold
Reset by pressing "4" and "5" simultaneously.

Make sure that SP (Set Point) is at a temperature 1 \(^\circ C\) below that of the sample, and make sure that (Output Power) is at "0". Start the power supply and the heating program (2 \(^\circ C/\text{min}\)) by pressing on the button 2, ">II". Write down the values of \( T \), \( F \), \( Q \) every 5 degrees starting from 50 \(^\circ C\). During the temperature variations, make sure the "electrode sample" distant remains constant, by moving the micrometric screw in such a way for the demodulator to always be set to the same frequency (Tuning on 0)

c) **High vacuum. Procedure:**

When running at full speed, the turbo-pump should never be in direct contact with a pressure higher than 0.05 mbar. Therefore, we proceed as follows:

a) Empty the chamber \( (P < 0.05 \text{ mbar}) \) using the rotary vane pump (VV switch, valve 2 open, measure pressure using Pirani gauge TM2)
b) Pump in the turbo-pump \( (P < 0.05 \text{ mbar}) \), (valve V3, measure using TM1).
c) Start the turbo-pump and use the rotary vane pump to pump “behind” the turbo pump.
d) If the pressure inside the chamber is still < 0.05 mbar, then open the valve V1 and the pressure should quickly fall from 0.05 mbar to \( 10^{-3}, 10^{-4} \text{ mbar} \) (measured using PM).

**Stopping the setup**

Turn off the oven’s power supply. Set the excitation amplitude back to zero. Using the micrometric screw, move the electrode approx 1 mm away from the sample. Turn off the power supply of the TECTANEL 1000 and the oscilloscope.

Close the valve V1. Turn off the turbo-pump, close the valve V3, turn off the vane pump, and turn off the main switch.