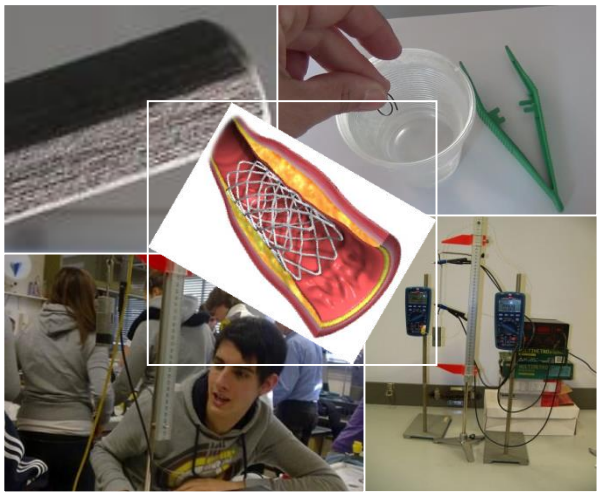
Shape memory alloys

Version: 12/06/2015



In nanosciences, the so called **smart materials** play a very important role. They are materials whose structure or composition has been specifically designed to induce properties which can be tuned and controlled in response to external changes in order to achieve specific goals. Smart materials integrate within the same device both a sensor, probing and measuring some kind of property in the surrounding environment, and an actuator, e.g a mechanical or electrical device producing a specific action in response to the sensing. The ensuing idea is that in principle it is possible to design new materials which meet strict technological requirements by controlling matter nearly atom by atom. A very important section among smart materials is represented by **memory shape metals.** In these alloys a slight temperature change or a mechanical strain can induce **changes at the macroscopic level**: shape, reflectivity, color, elasticity, electrical resistance, acoustic damping, etc. And, most important, once the material has been deformed, upon heating it goes back again to a specific pre-determined shape, whatever the deformation. This stunning effect is due to a modification of the crystal lattice, a solid-solid **phase transition**; called **martensitic transition.**

Contents

DISCLAIMER: The experiments described in the following document employ equipment and materials which need to be used according to MSDS specifications and according to specific school safety rules. Personal protection must be taken as indicated. As with all chemicals, use precautions. Solids should not be inhaled and contact with skin, eyes or clothing should be avoided.
Wash hands thoroughly after handling. Dispose as indicated. All experiments must be conducted in the presence of an educator trained for science teaching. All experiments will be carried out at your own risk. The entire NANOLAB team assume no liability for damage or consequential losses sustained as a result of the carrying out of the experiments described.

Notes

This Teacher Guide describes the experiments on memory shape alloys which you can find on Nanolab website www.nanolab.unimore.it/en/ at this page

Home > Labs > Memory metals

In addition to the detailed description of both the set up and the actual experiment implementation, the guide offers examples on how and where to link the experimental protocols to high school science curricula, links to background materials, tips on buying samples or any other equipment which may not be ordinary in school labs. Activities are numbered (1, 2, …) and match the corresponding experiments at www.nanolab.unimore.it/en/

On the website www.nanolab.unimore.it/en/, you can also find video guides, student lab sheets, presentations for classroom use plus an extensive collection of background reading for teachers.

In this guide the following symbols are used.

Qualitative demonstration. These experiments are particularly easy and need very little and simple equipment. They are suitable to be used in the classroom also outside the lab.

Quantitative experiment. These experiments involve data acquisition. The number of Erlenmeyer flasks states the difficulty level.

Safety tips, either regarding people or equipment (tools, samples).

Technical notes: technical tips, suggestions on possible alternative implementations.

It's possible to employ **electronic devices ,** such as smartphones and tablets, to collect data .

Use of **computer simulations** of the observed phenomena/ virtual experiments

Didactical notes. Teaching tips and didactical analysis

QR codes make the lab videoguide page, or alternatively videos of data sampling, accessible through tablets and smartphones

INTRODUCTION

Big idea #3: *Structure and functionalities*

The nanoscale structure of a material fully determines its characteristics and functions. Therefore by specifically designing and appropriately modifying the structure and composition of matter, it is possible to obtain properties which can be modulated in a controlled way, through external stimulus and for a specific purpose

1 Linking to the curriculum

The activities on memory metals are particularly suited to be linked to traditional curricular topics supplementing and completing them in a new perspective, namely matter studied at the nanoscale, opening the way to interesting interdisciplinary connections between chemistry and physics.

Below you will find suggestions on possible didactical paths

a) **Phase transition –** Shape memory effect in Nitinol is a quite unusual example of phase transition (solid to solid). Students are therefore urged to think about the real meaning of "phase" and "phase transition" as atomic structure modifications in the material with a clear counterpart in the macroscopic properties. The topic can be further researched comparing and contrasting with classical phase transitions (such as water/ice): isothermal VS non isothermal transitions, influence of stress/pressure on transition temperature, etc.

b) **Thermal expansion -** Common metals on heating expand, therefore exhibiting a positive thermal expansion coefficient. Different behaviors, such as those shown by a few metal alloys, are seldom described in standard Physics courses: Invar for instance has a zero expansion coefficient, while on temperature increase (through phase transition) Nitinol contracts. The explanation for this counterintuitive behavior has to be found at the microscopic scale.

c) **Electrical resistance –** The behavior of Nitinol electrical resistance with varying temperature greatly differs from usual metals. The origin of this apparent anomaly is once again to be found in the particular crystal structure thus naturally favoring the introduction of a quantum model for metal conductivity although only at a qualitative level.

d) **Sound transmission** – Sound transmission is strictly connected to the material structure of the propagation medium. Austenite phase in Nitinol is characterized by order and regularity with the strong simmetry in the crystal lattice favoring sound propagation. In Martensite phase, on the other hand, the less marked crystal simmetry and the differently oriented domains greatly impair the propagation of sound waves producing a thud and also a change in fundamental frequency according to temperature.

It is also possible to use this module in a context of **Inquiry Based Science Education** to promote experimental skills since the very first years. From this point of view it's not so

important to delve into specific topics but rather to focus on working modality. The use of a material such as Nitinol, with many appealing and counterintuitive properties, apparently challenging school linear physics, is particularly suited to this specific aim.

For a general introduction on memory metals see

Home > Labs > Memory metals > Background reading

2 Module guide

First of all students should be introduced to a few qualitative aspects of Nitinol, namely **shape memory** and the observation of the **activating temperature**. Even while carrying on this very simple demonstration it is possible to make them reflect about the actual meaning of **phase transition**. (exp.1)

As a second step more properties changing due to phase transition may be observed. Sound transmission and its **damping** plus the **shift in fundamental frequencies** are specifically investigated**,** both aspects being directly related to the change of the material inner structure. For the first time pupils will observe the **hysteretic behavior** which will be recurrent in Nitinol phenomenology. This represents a great opportunity to introduce a whole range of hysteretic behavior phenomena, the most known being magnetic hysteresis. (exp.3)

The apparently counterintuitive contraction of Nitinol spring calls for a clarification of the real meaning of the **thermal expansion coefficient.** Examples where such an index can exhibit either zero or negative values can be shown. The study of elongation versus temperature brings out a new hysteretic behavior and allows for further comparing and contrasting with classical phase transitions such as the solid to liquid transition of ice transforming into water. In Nitinol the process is not isothermal, while the influence of the applied load on transition temperatures reminds of similar influence of pressure in classical phase transitions. (exp.2 & 4)

Furthermore heating a Nitinol "spring" in a controlled way through Joule effect, offers the opportunity to monitor **electrical resistance** versus time and temperature respectively. It's therefore possible to highlight differences with what happens in common metals and outline the efficacy and importance of this physical parameter as a fine probe of the material microstructure. According to quantum theory of metal conductivity, resistance in a perfect crystal is zero and progressively increases according to structural disorder. During the phase transition from Austenite to Martensite Nitinol should therefore exhibit an istant of maximum disorder. Actually a peak in resistance is experimentally recorded! Last but not least it's possible to appreciate a change in resistance due to the varying mechanical stress applied. This is a key idea since it offers the opportunity to introduce pressure dependence of metals electrical resistance, a well-known fact but quite difficult to experiment first-hand in a school lab owing to the extremely high pressure needed. (exp.2)

Finally the last activity is dedicated to the use of Nitinol as ***muscle wires***, extremely thin niti wires which find wide employment mainly in **microrobotics.** Millimetric contractions are conveniently amplified and the wires exert notable forces acting at the same time as both sensors and actuators (exp.4)

EXPERIMENTS

1 – Shape memory effect

Shape memory alloys can be introduced with the following, mainly qualitative, demonstration. It is however possible to have students working in a semi-quantitative way even within this simple experiment.

Lab goals

- Demonstrate the "memory shape" effect
- Induce Martensite/Austenite transition through temperature change
- Determine activating conditions for the transition
- Demonstrate Nitinol retraining

What's to be observed

The experiment shows how Nitinol wire, which has been deformed at low temperature, regains its original shape on being heated. It will also prove that it is possible to give a new "memory" to the wire by retraining its shape at high temperature.

Equipment (for one working group only)

- Small piece of Niti wire (3 - 4 cm long) or, alternatively, a Niti spring
- pliers
- beaker with hot water (approx. 90 °C)
- temperature probe
- candle
- lighter

Link to the videoguide

Read the QR code on the right or go to the page

Home > Labs > Memory metals > 1- Shape memory effect > Videoguide

Background reading

Home > Labs > Memory metals > Background reading

Experimental protocol

A - Setup

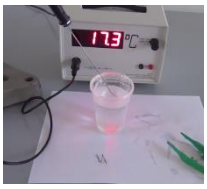
Clip the Nitinol wire in long short pieces (3-4 cm). For each group you also need a clear plastic glass or any other clear vessel and pliers to pick the wire out. (BE CAREFUL! Burning hot water!)

B – Shape memory

Fill the glass with hot water (80°-90° C). Bend the wire in the desired shape and dip it into the water. Make use of pliers to take it out again. CAREFUL: protect your eyes with googles! When water is very hot the wire will suddenly spring back as soon as it reaches the water surface or comes in contact with the hot vapour!

Careful! Burning water! Protect your eyes!

C – Activating temperature



With a temperature probe you can upgrade the demonstration to a quantitative investigation. You may estimate the minimal activating temperature by running the cycle again and again as the water cools down. At a specific temperature only a partial regain of the shape will be observed. This is due to the fact that the different martensitic domains will transition to Austenite phase at different temperatures. The phase transition Austenite ⇔ deformed Martensite in fact is not isothermal but rather takes place through a whole range of temperatures. With further decrease in temperature shape memory will not work any longer and the wire will be left deformed. When water temperature drops below a specific value the shape recovery mechanism will not work any longer. It is therefore possible to reach a first estimate of the activating temperature.

D - "Retraining" shape memory

It is possible to "retrain" the wire memory. Model the Nitinol wire in the desired shape (for instance a V or a S) and expose it to a very high temperature source such as a candle flame. At first the wire will try to spring back to its originally memorized shape exerting a very strong force. Be aware of this and hold the wire extremities tightly in place with pliers. Thirty seconds should be enough. However after a short while you will distinctively feel a relaxation in the wire: this is the unmistakable sign that the new shape has been memorized! Leave the wire to cool down before testing it and then repeat step B. Bend or straighten back the wire, then dip it into the hot water: it will now regain the "new" memory.

Careful: burning flame!

Do not expose the wire to the flame longer than the suggested time or you will damage it permanently !

If your aim is purely demonstrative you can use hot air coming from an hairdryer rather than hot water. However in this case it is almost impossible to reach an estimate of the activating temperature.

2 – Phase transitions: resistance, elongation

The topic can be developed further through the following quantitative experiments making use of a NiTi spring *(one-way smart spring)*[1]. Of course the same protocols are suitable to be used also for purely demonstrative purposes.

Lab goals

- Introduce an example of *smart material*
- Revise the meaning of "phase". When can you say that a material undergoes a phase transition? Which properties change?
- Observe phase transitions which are quite different from the usual ones (solid to liquid; gas-to liquid, etc…): Nitinol offers an unusual example of solid to solid phase transition which is rarely observed in a school lab.
- Understand that changes at the atomic level within the crystal lattice induce modifications in the macroscopic properties of a material.
- Link the experimental observations to the atomic scale behavior.
- Perform a quantitative study of Nitinol transition phases induced by a change in temperature and/or the applied mechanical stress .
- Introduce the quantum model for charge transport in metals. (Advanced)

What's to be observed

The experiment analyzes the behavior of a deformed Niti "spring" during phase transition from Austenite ⇔ to Martensite and vice versa as both temperature and applied load vary.

Specifically it is possible to

- study the counterintuitive behavior of the Niti spring which, on heating, contracts.
- analyze the dependence of the spring elongation versus temperature dependence (throughout the phase transition)
- observe the resulting hysteresis and study its dependence, together with that of the transition temperatures, as the applied load changes

(optional)

- analyze the dependence of the spring resistance versus both temperature and mechanical load dependence
- assess the efficiency of the Niti "spring" as actuator.

[1]See "Finding materials and equipment" at the end of this guide

Equipment (for one working group only)

- one way Niti spring
- mass holder + 50 gr masses (300 - 400 g total)
- power pack
- multimetre [2] - as voltmetre
- multimetre with thermocouple
- 4 electric
- connecting cables
- computer
- lab bar + horizontal arm
- chronometer (optional)
- video camera
- screen or white sheet (A4 minimum)
- ruler or video analysis software
- a small piece of thermo-shrinking plastic (optional)

Link to the videoguide

Read the QR code on the right or go to the page

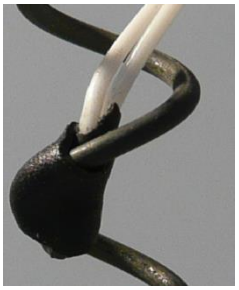
Home > Labs > Memory metals > 2- Phase Transition : resistance, elongation/contraction > Videoguide

Background reading

Home > Labs > Memory metals > Background reading

Experimental protocol

A – Setting up the temperature probe

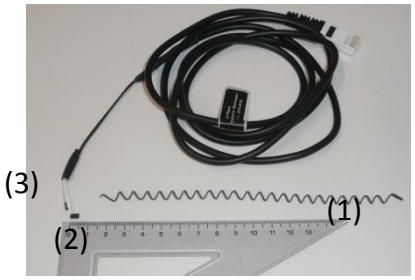
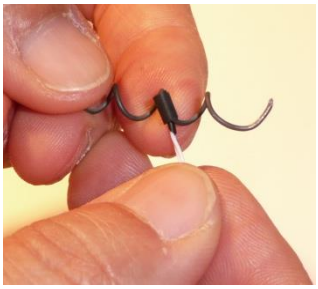
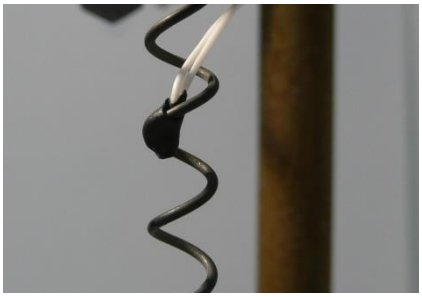


Cut off a small piece of thermo shrinking tube (approx. 5mm long). Insert the spring wire into it and slip the tube to the desired position (preferentially one of the two ends of the spring). Slip the thermocouple probe inside the tubing and make it adhere tightly to the spring surface. The rise in temperature produced by Joule effect the first time that current flows through the spring should be enough to make the plastic shrink and fix the probe.

Alternative modalities for fixing the probe to the spring should be able to resist to temperatures well over 100°C and still should not significantly dissipate heat. The shrinking tube dissipates very little and therefore doesn't impair measurements. However if the probe is too stiff, as the spring shrinks and wrings the plastic may break apart.

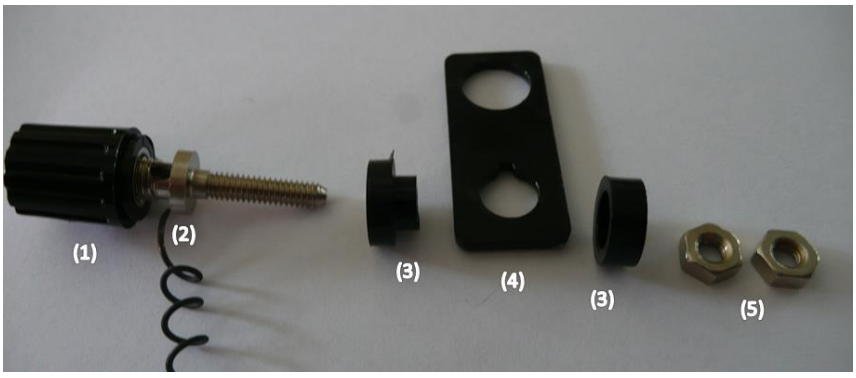
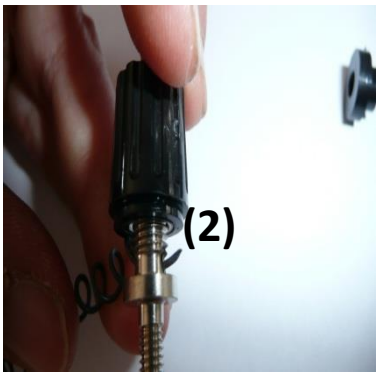
Always keep in mind that the probe measures **surface temperatures,** not the metal inner ones

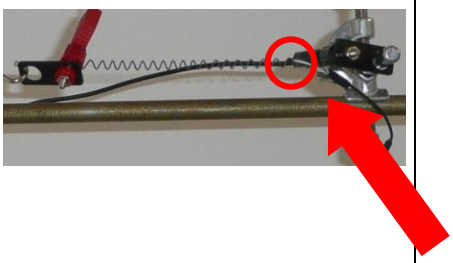
[2]Instead of two multimeters it is possible to use voltage and current probes for on line data acquisition. The circuit scheme doesn't change.

		
Slip the spring (1) into a 5 mm long piece of thermo-shrinking plastic tube (2).	*Insert the probe tip (3) inside the tube: try to keep it as parallel and as strictly in contact as possible with the spring.*	*Careful: the probe tip should not stick out of the tube. The heat produced by the spring through Joule effect when current flows should be enough to make the tube shrink and tighten around the probe.*

B – Setting up the spring for circuit insertion

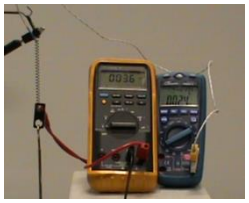
Hang the spring and load it with masses. Be careful to keep it electrically insulated from the metal hanger when current flows through. See the following scheme for a possible assembly.

	
In any electronic shop you can buy two plugs for each spring. Disassemble the plug: head(1) and screw (2), two plastic rings (3), two bolts (5). You will also need two double holed slates of plastic (4).	*Unscrew the plug head in order to see the hole in the screw body. (2). Straighten one end of the wire and slide it into the hole slightly hooking it for a better grip. Tighten the screw back.*

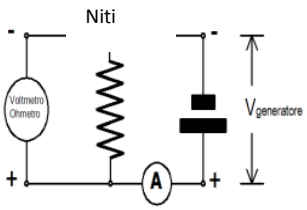
		
Fix the plugs at the two ends of the spring.Put the screw through one of the plastic plaque holes (3). Fix it up with the two bolts (5). Use the second hole either to hang the spring to the holder or to hang the mass holder to the spring.	*Alternatively the plaques can be made out of wood or aluminium. In case of a not insulating material. Be careful not to let the plaques in contact with the metal screw.*	*The spring is now ready. See the small piece of thermo-shrinking plastic used to fix the temperature probe to the spring circled in red.*

To save time give to the students the spring already equipped with the plugs and elongated. However tell them to use in the test the same mass you have used for elongation otherwise the corresponding hysteresis cycle won't close. Each time you change mass it's highly suggested to make a rehearsal cycle first, without data collection . This is why in the student sheet pupils are asked to run the first cycle just observing .

C – Building the circuit



Connect the spring and the power pack. You will read the current intensity values directly from the power pack display. Insert a multimeter in voltage modality (parallel connection). See the circuit scheme on the right. The spring temperature increases in a controlled way due to Joule effect. the spring has to be inserted in series to the power pack while the multimeter as a voltmeter in parallel. Detailed instructions on setting up the circuit may be found in the students sheet.



According to pupils level in circuits knowledge you can either instruct them step by step or let them completely free. Accordingly you will decide what to insert in the students sheet: circuit scheme, circuit explanations or just the request to have current flow through the spring and monitor both V and I. The sheet is editable .

D – Video recording

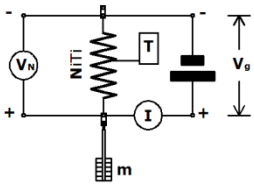
The variables to monitor in this experiment are temperature T, elongation L and voltage V. If you are not using on-line data acquisition the best way to keep control of all these parameters at the same time is to shoot a video with either

a videocamera or a photo camera with automatic multiple shots). Since the shrinking process is extremely fast it's almost impossible to measure L with a ruler in real time. However thanks to video recording it will be easy to select the pictures on which measurements will be taken either manually or with a specific tracking software.

Pack the equipment tightly so it will result all within the same video frame. Put a screen or a white sheet behind the spring. With colored tape make a visible spot somewhere, for instance on the lowest weight attached to the spring. This will be used as a reference mark by the video analysis software in tracking mode. Check that both the power pack and the multimeter display digits are clearly visible and can be easily read on the video. (It is highly suggested to make a video recording test before running the real experiment: too little light, light shining on displays, wires running in front of displays and so on may all prevent good readings). The best measuring instruments for video recording are equipped with red digits displays.

Make use of either a tripod or a fixed stand for the video camera.

A photo camera is a valid alternative too provided it is a multiple shot one (at least 20 shots in a row). Many cameras have automatic shots but are not able to shoot more than three in a row. To record the whole shrinking process you need at the very least 20 shots, one shot per sec. While cooling is usually a slower process and you can shoot manually at longer intervals. For instance you may decide to take a shot each time temperature decreases of 1°C. You can also ask pupils to shoot videos with their cells. In addition to all previous suggestions ask the students to check that the video format is compatible with the video analysis software and in case find a video converter software .[3]



Choose an initial mass (typically 250 gr) and hang it to the spring with the mass hanger. Switch on the power pack and let a constant current flow (suggested intensity between 2 - 2,6 A). The spring will begin to heat and consequently to shrink. Shrinking will be completed in a few seconds and final temperature may reach over 100°C. To get resistance data during cooling turn down the current to 0,5 A but don't switch it off altogether!

If you have enough time it would be quite interesting to repeat the cycle with different applied masses. However upon changing the mass to cycle once before you take data.

[3] For instance . http://www.freemake.com/it/free_video_converter or AnyVideoConverter

Read carefully the data from the retailer to avoid damaging the spring with excessive current or mechanical load. Hints reported here refer to springs bought at www.mindsetsonline.co.uk.

- ***Never exceed 3 A in current and 500 g in mass!***
- ***Take care not to elongate the spring further than 10 cm!***

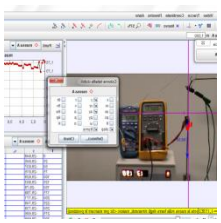
On cooling it's also possible to read the resistance directly with the multimeter as ohmmeter. However ohmmeters you can usually find in schools will not give out measurements beyond one tenth of an ohm. This is why we suggested in both cases an indirect measure of R=V/I. Once the spring temperature has reached room temperature, switch off the power pack.

***Temperature T, current intensity I, voltage V**: are all read straight from the instruments; **Resistance R:** is calculated as V/I; **time t**: is the interval between two shot or, alternatively, is read on the vide timeline; **Lenght L:** measured from the pictures.*

One of Nitinol major issues, as application developers well know, is its low reproducibility. This is one of the reasons why (in addition to the obvious advantage of saving time) all data are collected together within the same experiment instead of being collected with different experiments. Although an apparent drawback this actually is a unique opportunity for students to work in inquiry based modality. Examining the whole range of collected variables they will decide on their own which variables should be correlated and how to refine their investigations. It wouldn't be surprising if they were able to find new correlations others than those pointed out by the teacher.

The "low riproducibility" issue must be taken into due consideration also while interpreting results. The collected data strongly depend on the spring "history", so that , even if different groups work with perfectly identical springs, perfect coincidence in data comparison should not be expected.

E – Video analysis



Use a video analysis software with automatic tracking functionality, such as Tracker[4], to monitor the spring movements. For further analysis and investigation paste all data and synchronize them in an electronic sheet. On the website you can find a tutorial of Tracker software (It's in Italian but very visual - EN tutorials can be easily found by googling "Tracker"). There are essentially two modalities for data collection. In both cases the use of video analysis software is highly recommended. To practice you may download use the video at the website

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[4] See "Finding materials and equipment"

1- The first modality is quite simple and "low tech ", even a bit boring! It can be used with very young students to reinforce scale reduction concept and the use of proportions.

2- The second modality is "technologically more advanced" and it will be certainly appreciated by students, mostly by those who enjoy playing around with technology. Moreover learning to use Tracker to note down instant position can be extremely useful for future applications, mainly in mechanics. Tracker however provides only position, speed and acceleration. Other variables such as temperature should be read from the video.

MODALITY I – Manually

Once the video or the photos have been downloaded on the PC the measure of L can be obtained directly from the screen with a ruler, or through the cursors of image manipulation software. After calibrating with a known length, such as a tape strip or a 10 cm long stretch highlighted on the ruler beside the spring, the spring length is measured at fixed time intervals (video) or shot by shot (photo)

Careful: a ruler set in the background will be clearly visible in photos but it may result out of focus in a video and utterly useless for calibration. In automatic mode in fact the focus is on the spring and its movements rather than the still elements around it. To avoid all this just switch to" manual " .

In case it is also possible to print out photos and work on paper. See "Typical results " at

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MODALITY II – Tracker or other video analysis software

Data are collected through video as in the first modality but the elongation is obtained with the video analysis software Tracker. For a dedicated tutorial see at the above page under the voice "laboratory guides"

Tell students to keep collecting data till temperature reaches the equilibrium with the external environment even if the spring doesn't elongate any longer.

After saving video/photos on the PC students will be able to work at homeHowever it is highly recommended that they work in groups. Particularly if data acquisition is manual tell the students to collect the values of all kind of variables and store them in a spreadsheet for future use.

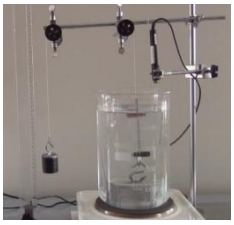
MODALITY III *On-Line data acquisition*

A third modality is online data acquisition. Tracker is used as in modality II. The multimeter with the temperature probe is substituted by an on-line temperature probe; while the voltmeter with a voltage on-line probe, both connected to the computer interface. It's extremely important to synchronize both probes and the tracking when pasting data on the spreadsheet [5].

[5] Data collection rate: 1 per sec; Tracker step: 25 frame per sec.

In our case data have been collected with Vernier sensors and data logger Lab Pro with Logger Pro software .

G –Heating-cooling the spring in thermal equilibrium steps



The heating process can be achieved through a succession of tiny steps ensuring thermal equilibrium with the environment. Put the spring into a thermal bath. The whole process is much slower than heating by Joule effect and then cooling in air but it's also far more precise. This method can be very useful if you want to investigate further the hysteresis effect in the spring elongation/contraction due to temperature change and have no interest in measuring resistance.

Equipment (for one working group only)

- one way niti spring
- 400 gr mass
- beaker (5 lt)
- hot plate
- 2 pulleys
- temperature probe
- lab bar + horizontal arm
- chronometer (optional)
- video camera
- thin rope (1 m)
- ruler or video analysis software

Alternative to the beaker you can use any kind of pot. However the effect won't be so spectacular if you can't see through a clear container. From a quantitative point of view this is not important since the recorded movement is not the spring one but rather those of the external weight connected to the spring through the rope and the two pulleys.

Data analysis

Collected data range over a wide variety of variables. Students may analyze the data deciding to match variables in different ways according to the experiment goals.

Elongation versus temperature

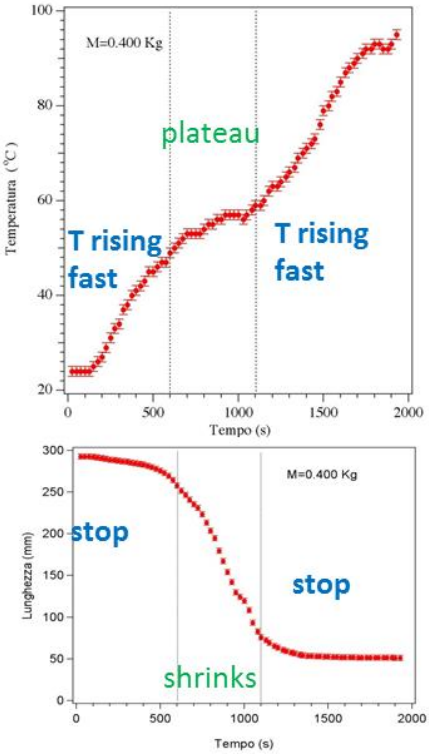
If you want to set the stage for *inquiry based* methodology first invite students to examine which are the important variables for a quantitative description of the phenomenon**.** If, on the contrary, you'd rather opt for a more guided working modality, tell them straightly to focus on elongation and temperature. At first it's much better to analyze only the data collected during the heating process, leaving the comparison of heating and cooling process for further investigation.

To plot elongation versus temperature, you may suggest the following steps :

1 – plot temperature versus time

2 – plot elongation versus time

You should get two graphs like the following ones



Comparing the two graphs it is possible to notice that at the beginning, when the spring is not yet moving, temperature is already quickly rising. While during the shrinking process temperature still increases but much more slowly. Then once the spring has reached maximum shrinkage it rises again much faster.

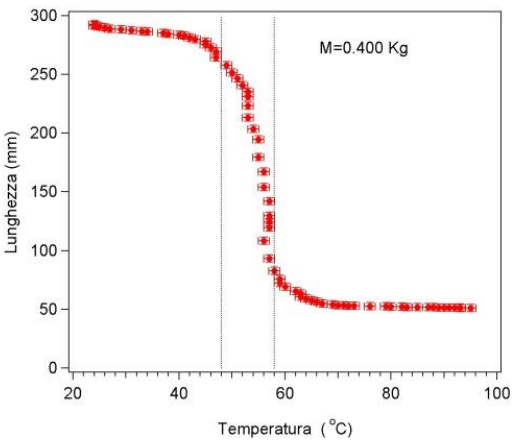
This behavior (see above graph on the left) reminds of the isothermal behavior (with a typical temperature long step) shown by the classical phase transitions which should be well known to students (melting, vaporization, etc.). In Nitinol the transition is not exactly isothermal, however it is possible to point out a specific temperature at which the transition begins and one at which it is completed (respectively A^s and A^f , see background reading). If the acquisition rate is fast enough (at least 1 sample per second) it is possible to notice that during the shrinking of the spring, temperature increases by stops and go with a succession of tiny landings visible on the graph. This is even more evident at lower current (1,9-2 A). At macroscopic level this phenomenon can be related to the fact that the transition from Martensite to Austenite is only locally isothermal, that is to say every martensitic domain within the material has its own transition temperature dependent on the local conditions of the domain itself. The presence of defects, local stress, etc… For further research see the background reading and the videos mentioned below.
Short video clips with microscope images of martensitic transformations will be very useful to give an idea of what's really happening at microstructure level. It may be appreciated the "stop and go" movements, the de-twinning, the augmented disorder of the martensitic phase. It is possible to revise experimental data and try to interpret them according to the videos

a. http://www.youtube.com/watch?v=qZsozvf1pD8 *Courtesy of DoITPoMS, The University of Cambridge. Released under Creative Commons Attribution-Non-Commercial-Share Alike license*
b. http://www.youtube.com/watch?v=OQ5lVjYssko&feature=related *University of Tokyo.*

An alternative to the videos is the following simulation http://www.smaterial.com/SMA/simulation/md_simul/MD_start.html

3 – plot elongation versus temperature.

You will obtain a graph like the one on the right.

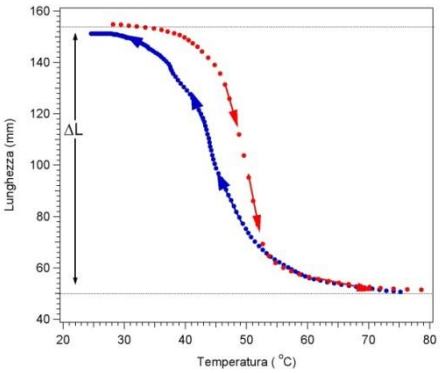


In this graph it is even more clear that the

transition takes place in a restricted temperature range with "stop and go" shrinking movements. It will be therefore reinforced the idea of the initial demonstrative activity, e.g the most important driving phase transition is temperature.

4- Ask students to give an **estimate of both initial and final transition temperature**. The best way to quantitatively estimate the two temperatures is the **tangent method**, described in the following paragraphs.

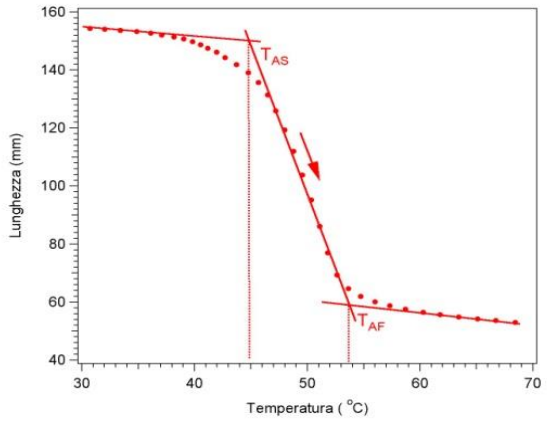
If you intend to discuss the hysteresis cycle, ask students to plot elongation versus temperature also during cooling process. In the picture on the right in red the data collected upon heating while in blue data collected upon cooling.

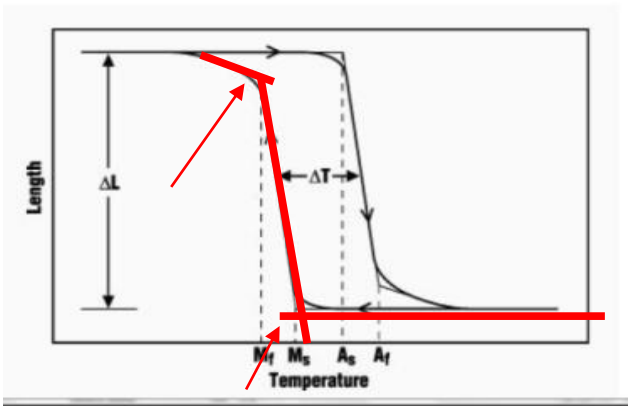


It's quite evident that the two graphs do not overlap, which means that the two processes of heating and cooling are not exactly inverse ones. In particular the initial and final transition temperature are different whether in heating or in cooling. This is a typical hysteresis phenomenon, similar to what happens in magnetic materials. The experiment can be used as a starting point to research further this class of phenomena

Measures have to be carefully taken and if necessary repeated. If the probe is not in complete contact with the spring or in case of excessive heat dissipation through the connecting system relevant errors may occur. Even the research for transition temperatures with the tangents method may not be an easy task and could be affected by large errors.

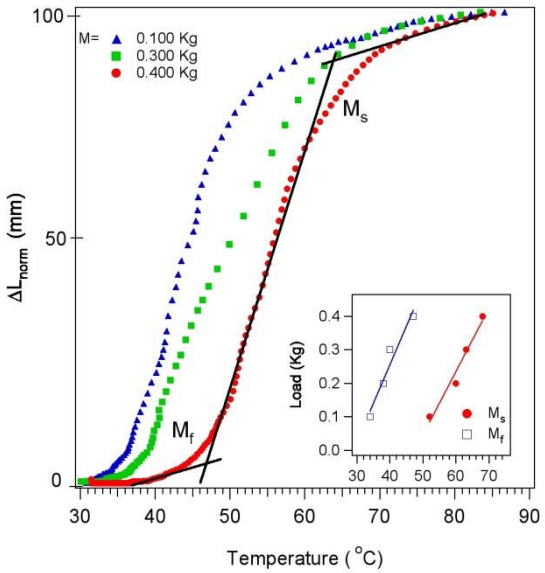
Tangents method for evaluation of phase transition temperatures





To give a quantitative estimate of initial and final transition temperature, the tangents method may be applied. The key idea is to approximate the experimental data curve (elongation VS temperature) with a polygonal. These are the main steps:

- Draw the tangents to the different parts of the curve (in red in the right picture above).
- Graphically find out the tangent intersections points (see arrows, same picture).
- The abscissa of the intersection points give the transition temperatures (in the graph on the right it is possible to see all the four temperatures).
- Either trace the tangents on an already printed out graph or use the specific functionality of any advanced data elaboration program (picture on the left).

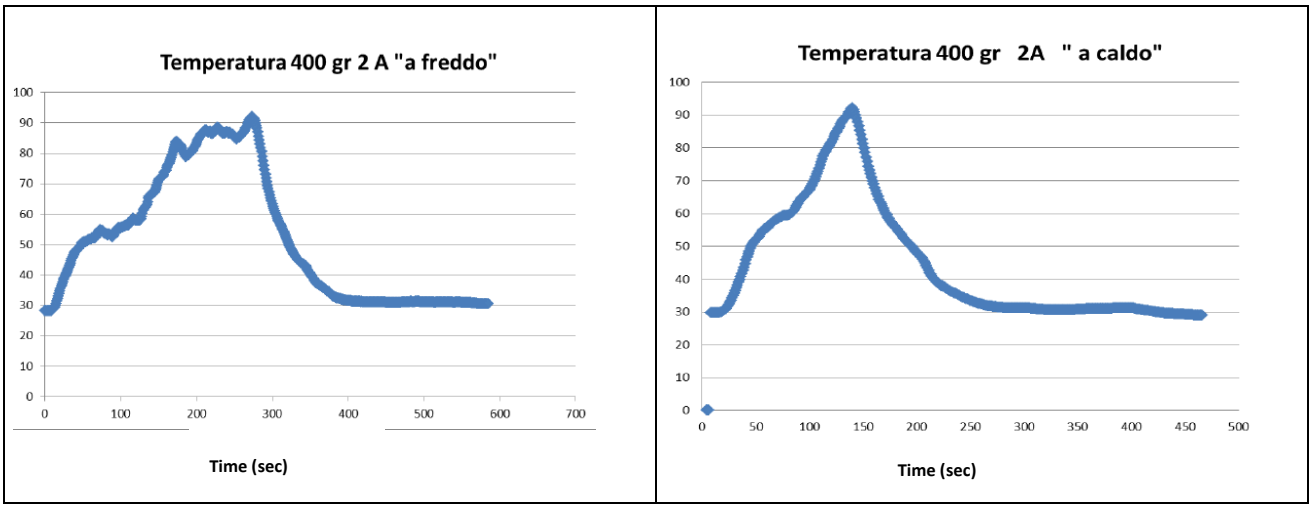


5 – If there is time enough to **repeat the cycle with different masses** it will be noticed that varying the applied mechanical stress, will result in a change of transition temperatures: the bigger the masses the higher the transition temperature. See inset picture Data collection during cooling process [6] may be easier and less tricky.

To research further (advanced level) and to prove the higher regularity of the cooling process over the heating one it is possible to run a cycle with the same mass applied to the spring starting first with a "cold" spring and then with a "hot "[7] one .

[6] It is advisable to work with the cooling process and related curves since this process is a succession of spring/environment equilibrium steps and therefore shows a more regular trend with respect to Joule effect heating. As an alternative use thermal bath.

[7] "Cold" – let the spring rest for 10' or 15 'in between each cycle. This guarantees that the cooling process is completed also within the material and that the domains have gone back to the original configuration.
"Hot"- the spring is cycled again immediately again after each previous cycle. When the external temperature seems to be back again to the initial equilibrium, actually the inside of the spring is still hot and the martensitic transformation not yet completed.



On heating started with a "cold" spring the curve appears much more jagged and irregular (left). Some of the energy (heat) is used to readjust and steer domains: actually small steps and landings in the temperature may be observed locally and sometimes there are even abrupt decreases although in a globally increasing trend.
Instead starting from a "hot" spring the curve appears much more smooth and regular. The inner domains are most probably still oriented according to the previous cycle and as a consequence the whole process is much faster and "smoother".
With higher currents the increase in temperature is much more regular and there's very little difference between heating starting from either a "cold" or a "hot" spring. From an applicative point of view (Nitinol springs as actuators) these are exactly the current intensities needed for an homogeneous and uniform operating of the mechanism.
Whatever the modality for heating there's no substantial difference in the cooling process.

At the end of this first part of data analysis it is possible to summarize differences and analogies between classical phase transitions and the new example offered by Nitinol which exhibits a transition with the following characteristics:

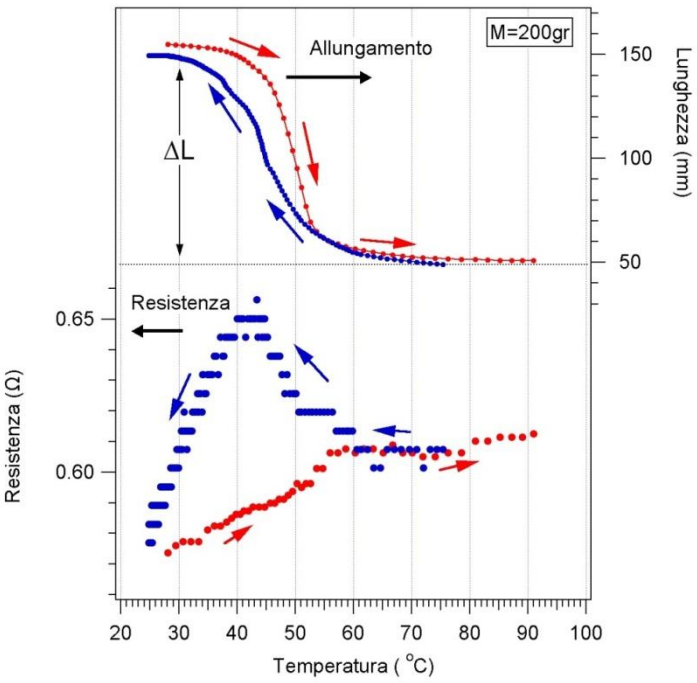
- ***Not* diffusive** *(displasive). Atoms rearrange themselves in a cooperative way in a new crystal structure with no chemical change. The atom movements are just tiny ones, there's no atom movement covering long distances.*
- ***Not isothermal**: there's not one distinct transition temperature but rather a range of temperatures for each phase. Moreover, differently from the classical case, in which for instance melting and solidification transition temperatures correspond, critical temperatures change according to the transition direction (hysteresis).*
- ***Athermal:** the amount of domains who have already transitioned into the new phase depends only on the temperature reached and not on how long is the isothermal permanence. In fact since no atomic migration is required, displasive transformations progress **independently from time.***
- *As the critical temperature of classical phase transition depends on pressure, the **temperature intervals** at the beginning and end of each phase **depend on the applied mechanical stress.** This is a directly proportional relationship.*

- ***Of the first order**, which means that heat is being released when the material transitions into Martensite phase, exactly as it happens in the passage from water to ice.*
- *The phase transition is highlighted by a set of **changes in macroscopic properties** such as resistance and elastic constant.*

Resistance in the different phases

Resistance is measured in indirect modality, by monitoring voltage and current and calculating R = V/I. In the heating process for a 250 gr mass the suggested current value is about 2,4 A, while on cooling the current is switched down to 0,5 A in order to significantly reduce the heating. Maintaining a small current offers the opportunity to measure resistance through the voltmeter; the small current value doesn't impair the spring cooling process and it constantly guarantees an acquisition sensitivity (the voltmeter one) which can easily reach the fourth decimal digit. The same multimeter in ohmmeter modality and with an open circuit would have a completely lower sensitivity (only one decimal digit).

One of the main goals of the experiment is to prove that resistivity can be a fine probe of Nitinol microstructure. Resistivity in fact indirectly allows to point out the changes taking place at crystal lattice level. Resistance is directly proportional to resistivity through geometric factors (length, wire section) which at first approximation can be considered not dependent on temperature (the spring length changes due to a change in shape during the transition, while the spring variation in linear length is negligible). Measuring resistance versus temperature offers direct info on the resistivity change during phase transition and such a parameter is strictly related to the actual degree of structural disorder. According to quantum model description, in fact, the electrons behave as waves and are not hindered in their propagation by a perfectly regular lattice. In other words, the interaction between the electron and the ions with a consequent increase in resistivity, takes place only if the crystal is not a perfect one. The main causes for "imperfection" are ions thermal vibrations and presence of impurities, or even possible edges and structural defects in the crystal lattice. As it was already shown, during phase transition such disorder is quite high, since the two coexisting phases form alternating orientations in domains. As a rule Martensite structure is more disordered than Austenite.

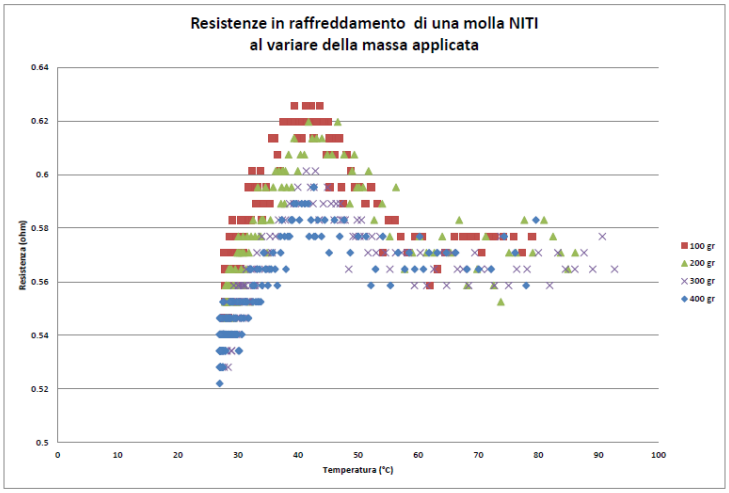


A sample of Resistance versus

temperature is shown in the picture. Below, you can see the spring electrical resistance upon heating (red I = 2,4 A) and cooling (blue I = 0,5 A). It is clearly outlined the hysteresis. Above you can see also the corresponding elongation versus temperature graph to facilitate comparison between R andcorresponding spring length.

The shape of the curve may change according to microstructure, however some common and recurring traits are clearly identified.

- R significantly increases upon heating within the temperature range in which the transition occurs.
- For higher temperatures resistance is almost constant.
- This global behavior drastically differs from the usual (small) direct proportionality between R and T as recorded in metals .
- The net variation in resistance during the transition (40°-55° approx.) can be associated to the presence in this phase of high structural disorder due to domain reorganization. Such an hypothesis is supported by the fact that on the contrary in the temperature range beyond transition (55°-70° approx.), although the absolute variation in T is comparable, resistance increases much more slowly (remember that the thermal coefficient of the metals resistivity is approx. 10^{-3}/°C thus comporting a variation of a few per cents for an increase in temperature of about ten degrees) .
- Finally during heating it is possible to notice a peak connected to an intermediate phase between Martensite and Austenite (rhomboedric phase).
- On cooling there's a very pronounced peak at the beginning of the martensitic phase due to the fact that this is a transition moment and the nucleation and formation of martensitic domains produces in the material a highly disordered structure with consequent high resistivity. Gradually the Martensite structure settles throughout the material becoming homogeneous. This brings to a decrease in resistivity, which progresses further due also to a drop in temperature.



- As we increase the applied mass, a progressive decrease in resistance is recorded, in analogy to the decreasing resistivity in metals under huge pressures.

Efficiency of the actuator

Nitinol springs are used both as sensors and actuators: if current runs through them (external stimulus), they shrink thus activating a predesigned action (such as traction) on the surrounding environment.

We may want to calculate the spring efficiency in the transition from Martensite to Austenite, while lifting the applied load. With the multimeter in parallel in the circuit it is possible to calculate the power output of the generator: input power $\mathbf{P_{in} = Vi;}$ the work done is $\mathbf{L = mgh}$ and the output power is $\mathbf{P_{out} = L/t}$. The transformation efficiency can be therefore calculated as $\mathbf{P_{out}/P_{in}}$.

Further research may include modifications in current I in order to modify the the contraction speed v of the spring. What's the relationship between I and v? How does efficiency vary?

3 – Phase transitions: acoustic properties, pliability

As already stated, different crystal lattice configurations induce different macroscopic properties in the same material. A careful observation of such properties is therefore a way to indirectly prod the micro and nanostructure, as it happened with resistivity. The investigation of sound transmission in Nitinol bars, which will be developed in this section, is particularly apt to a quantitative study .

Lab goals

- Reflecting on the meaning of "phase" in a material
- Understanding the effects of crystal lattice modifications on macroscopic properties
- Making a quantitative analysis of acoustic properties in Nitinol phase transition induced by temperature changes
- Linking experimental observations to atomic scale behavior

What's to be observed

At first a set of properties such as smoothness, reflectivity, pliability and, last but not least, sound transmission are investigated at a purely qualitative level in two Nitinol bars expressely made with two slightly different compositions so that at room temperature they will exhibit two different phases.

As a second step it's possible to upgrade to the study of **sound transmission** and **emitted frequency** in the same bar **with varying temperature**.

Equipment (for one working group only)

- 1 Ni51Ti49 cilindrical bar (lenght. 22 cm ; Ø 0,9 cm)
- 1 cilindrical NiTiCu10 bar (same dimensions)
- rubber hammer
- oven glove

- 1 cilindrical INOX[8] bar (same dimensions)
- 2 temperature probes
- computer
- Audio recording and analysis software[9]
- hot plate
- Glass pan Ø > 20 cm
- Pliers
- microphone

Link to the videoguide

Read the QR code on the right or go to the page

Home > Labs > Memory metals > 3- Phase transition: acoustic properties

Background reading

Home > Labs > Memory metals > Background reading

Experimental protocol

A - Comparing Austenite/Martensite properties in nitinol bars

Take two Nitinol bars A and M which are in Austenite and Martensite phase respectively at room temperature. You can compare and contrast their roughness, color, reflectivity, pliability and, most of all, acoustic transmission. To test this last property just throw the bars to the ground and listen to the produced sound: you will hear a ring (A) and a thud (M)

It would be great if one of the bars was specifically kept for this use and exchanged among groups. For the coming experiments it would in fact be better if the bar was not bent or deformed in shape.

Throwing the bars to the ground may result in bending which could be an issue in the following quantitative experiment. Although it's true that the bar can be almost completely brought back to its linear shape thanks to shape memory effect, however it is highly probable that it will not completely recover.

When the bars are thrown down record the produced sound with Audacity. Listen to the recorded file first at 44100 Hz and then in "acoustic slow motion" modality (Audacity allows to save at 8000 Hz): the result is amazing! You will find a sample of both files in the introductory lab page under "data samples".

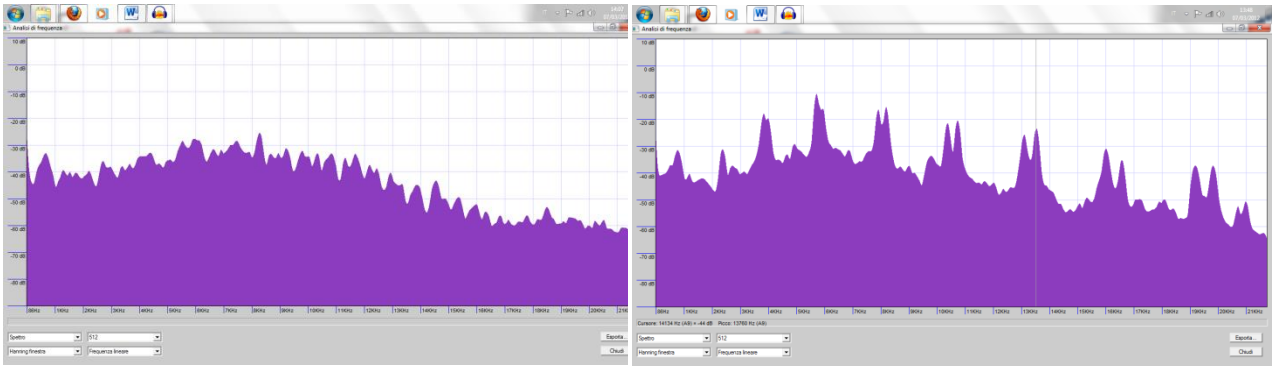
Bring to the students attention the fact that the two bars have slightly different compositions specifically because at room temperature one should be in Austenite (Ni51Ti49) and the other in Martensite (NiTiCu10) phase. To fully appreciate the effect that the phase transition plays on the same bar heat the Martensite to approximately 55 °C by dipping

[8] Any other metal is ok provided that the bar dimensions are similar to the Nitinol ones.

[9] See software free Audacity (http://audacity.sourceforge.net/)

it into warm water. The change should be very clear even at qualitative level.

When the two bars are thrown down to the ground the produced sound is a ring for Austenite and a thud for Martensite (listening to the recording of Martensite sound reminds of a wooden stick rather than something metallic falling). The sound spectra of the two bars highlights the difference: on the left Martensite and on the right Austenite. In Austenite there are recurring double peaks due to the rectangular section of the bar (if the section is almost squared you will see just one peak). The ratio between the peak frequency of each couple is directly proportional to the ratio of the bar section dimensions. The recorded sound is analyzed with Audacity (see step B and D below)



The ordered crystal lattice in Austenite allows the sound wave to go through the material without encountering any obstacle and therefore fast enough. The sound similar to a ringing bell as we would expect from a metallic object. In Martensite, on the other hand, the borders between regions with different orientations in the monoclinic structure are much less symmetric and cause vibrations to be reduced therefore smothering the sound. This naturally reminds of the walls of an anechoic chamber, especially designed to reduce sound reflection (left): its structure is very similar to the different orientations and irregular distribution of martensitic domains (right: picture taken from the microscope video).

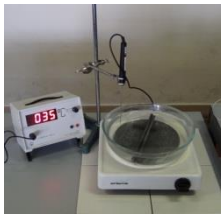
You may appreciate some similarities: From Wikipedia

From the video by Tokyo University

B – Audio recording setup

Download and install the freeware recording and audio analysis software Audacity (http://audacity.sourceforge.net/). Connect the USB microphone to the PC and make a preliminary test in the quietest room available whether your microphone is able to detect and record even the muffled sound of Martensite. Record 1' of "silence" and store the file: it will be useful in order to subtract and cancel the "background sound" from further recordings.

If you use a notebook detach it from the grid in order to limit to a minimum the disturbance coming from the "electromagnetic rumor". Position the microphone as far away as possible from the computer and as near as possible to the bar, in order to drastically reduce background noise

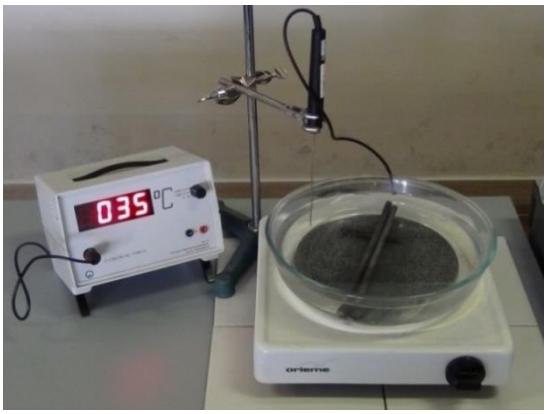


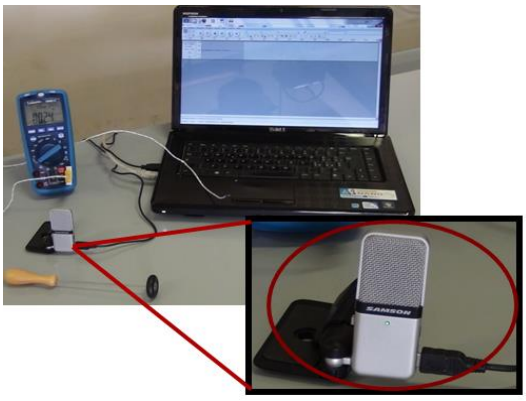
C – Sound transmission with varying temperature

Use two bars with similar shape and dimensions, one made out of Nitinol, the other out of iron. The iron bar is the "control" bar. The two bars are first brought from 0 °C (the temperature is reached by storing them in the freezer for at least 15') to approximately 98°C (in a thermal bath) and then cool down spontaneously. During both heating and cooling process the sound produced by the two bars will be recorded approx. every 5°C. Check the bar temperature, hold the bars firmly at a node[10], start recording and hit repeatedly in the middle with a small rubber hammer. at one end or in the middle. Try to hit regularly with the same intensity and at the same spot. Stop recording and save the file. Repeat such a procedure throughout the whole cycle (first heating and then cooling) at regular temperature intervals, say each 5 °C.

For an introduction to vibrational modes of a bar go to the following website (in particular the pages listed in the Reference at the end of this guide)

http://fisicaondemusica.unimore.it





[10] To identify quickly the position of the node make a sign with an indelible pen on the corresponding position: distance from the extremity = 0,224*L, where L is the total bar length.

The heating process from 0°C to room temperature will take place spontaneously. To reach higher temperatures however you will have to put the bar in a thermal bath. The two bars are immersed in a circular pot (they should lay horizontally and be completely covered by water).

With a temperature probe monitor water temperature: it will be heated up to boiling point with a hot plate. As they reach thermal equilibrium liquid and bar should be at the same temperature but actually when you take the bar out it will fast cool down also because of its relatively small dimensions and the high thermal conductivity of the material. The bar temperature is measured twice, just before and after having been hit. In between the hits the bar is stored back into the water.

Use pliers to extract the bar from the boiling water and a kitchen glove to hold it at the node.

The cooling process takes place naturally from approximately 95°C down to room temperature. The control bar made of inox undergoes the same procedure (just run it in parallel)

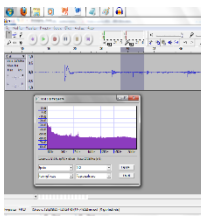
The bar is gripped in the calculated node position of its fundamental vibrational mode. This should ensure the reproduction of the free oscillation and dampening should be drastically reduced.

If the bar has a rectangular base be careful to grip always the same two faces (that is equivalent to dampening always in the same direction). The advantage of a rectangular base with a noticeable difference between the two dimensions is that the double peaks are reduced.

The choice to refrigerate the bar at 0°C is led by the desire to record the sound at temperatures near room temperature reached with a heating process . If the bar is taken out of the thermal bath around 25° C and hit at 20-22° C (due to thermal dissipation) this temperature has actually been reached on cooling and NOT on heating. While the difference is absolutely negligible in normal metals it may be of relevance in Nitinol, since the two processes are not completely reversible ones .

The uncertainty in temperature is quite high because the cooling process is very fast, particularly when the thermal gradient with the external environment is big. Moreover temperature probes are not so prompt after all. Always remember that the one you measure is only a surface temperature. In spite of all this a regular trend can still be observed.

D – Sound analysis and data treatment

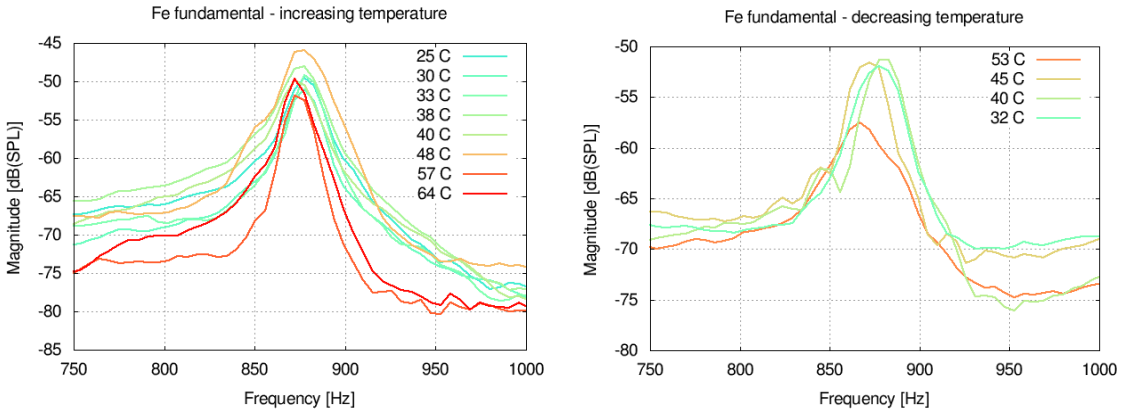


Obtain the sound spectrum with Audacity from each audio sample and extract the fundamental vibration frequency for each temperature. You can recognize it from the highest peak within the frequency gap that is in agreement with the theoretically obtained value. The comparison with the "silence" sound spectrum previously recorded can be very helpful to recognize and dismiss anomalous peaks, not produced by the bar. For a detailed description of the steps have a look at the tutorial in the following page.

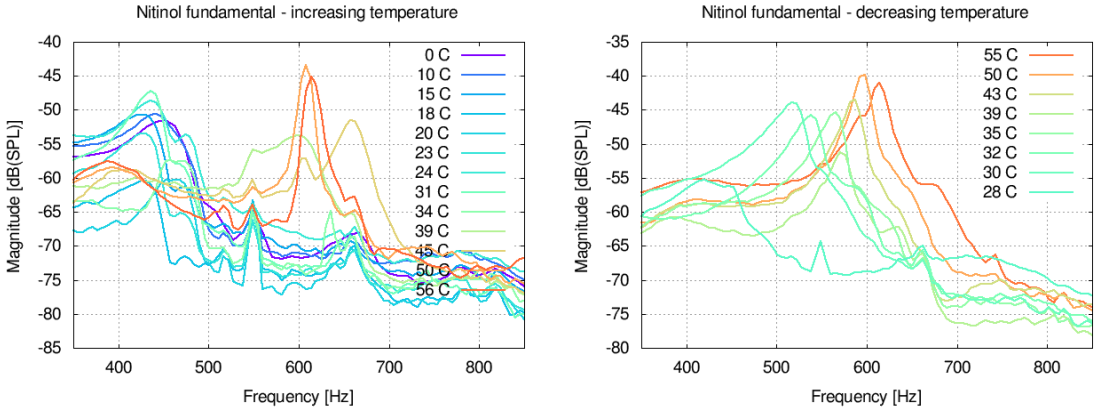
Home > Labs > Memory metals > 3- Phase transition: acoustic properties

Upon being hit the Nitinol bar it emits a not harmonic spectra and it is immediately

possible to observe the change in tonality depending on temperature. You will find an example at the end of the above link page under "data samples". You can also find a similar file for the iron bar. Listening to them in strict succession the Nitinol specificity will stand out clearly. A quantitative analysis is made only on the fundamental frequency. To obtain the maximum excitation mainly of the fundamental frequency the bar should be hit either to one end or right in the middle as it is firmly gripped in one node. On the contrary the iron bar doesn't exhibit any noticeable modification of the emitted frequency when temperature varies (the volume expansion/contraction is not such to influence density or elasticity significantly inside the considered temperature range). The frequency variation are all within the intrinsic width of the peak.



The Nitinol bar on the contrary shows a very clear shifting of the peaks as temperature increases together with a larger band width.

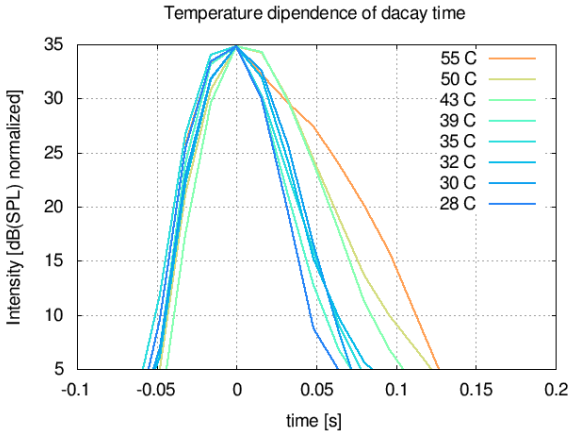


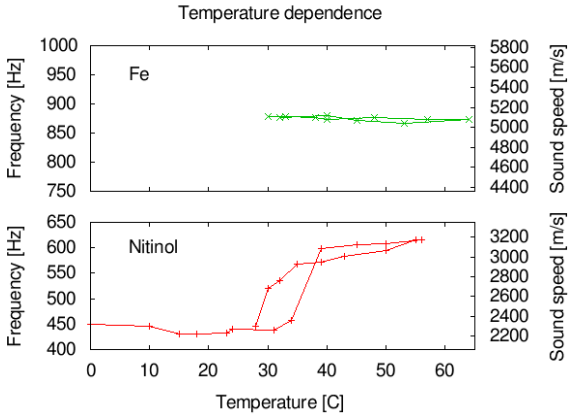
The fundamental frequency f thus measured can be used to obtain sound speed c in the material with the following formula valid for cylindrical bars where d is the diameter and L the bar length.

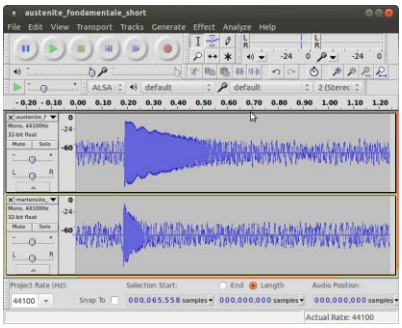
$$f = 0.890 \frac{d}{L^2} c$$

As we plot fundamental frequency versus temperature it's once again possible to observe an

hysteretic behavior in Nitinol. Nothing of this happens with iron. This specific result justifies the particular attention put in sound recording through a definite heating process at temperatures near the ambient one. In the graph you can also read the corresponding sound speed in the material.







Also the damping time in Nitinol is strictly dependent on temperature and it clearly highlights the muffling properties of mechanical waves typical of martensite phase at the lowest temperatures. Clearly in Martensite estinction is much faster. This property is exploited for instance in antisismic applications or in machinery. When damping time stretches it is a sign that Austenite is on the way. It is therefore clear that both intensity and quality of the emitted sound may offer precious info on the actual phase of the sample

E – Data acquisition by smart phone - alternative modality

A valid alternative to the previously described procedure for sound acquisition is the use of smart phone audio recording functionality. The microphones of smart phones are in fact suitable for high quality recording. Such recordings will be saved into *.wav* files and can be subsequently analyzed with Audacity software. Those wanting to use a smartphone for spectrum analysis as well, will find free apps on line[11] however they don't usually allow to save the results in a file. This is why Audacity is highly suggested for the analysis even in case it has not been used for recording.

The use of sound waves or ultrasound to test materials structure is no news. Sismic vibrations have always been exploited by geologists to investigate the inner structure of our planet. Nowadays the application of waves as an investigative tools ranges from research of inner defects in materials (it's widely known that Nitinol was "discovered"

[11] See "Where to find materials and equipment" at the end of the guide

thanks to the anomalous sound similar to a thud, produced by a falling bar. At first researchers thought it was nothing else than a faulty bar) to the investigation of bone age to prevent ostheoporosis. They may be used also to give an estimate of ice thickness or water salt content. There's a research branch specifically called 'acoustic thermometry' or 'acoustic scan microscopy'.

Many of these investigations are based on the fact that sound usually travels at different speed in different media. Sound speed C is in fact related to the mechanical properties of the propagation mean, mainly elasticity Y and density D, or rather, their ratio $C^2 = Y/D$. In a stiffer medium sound speed is higher but with the same elastic module in the denser medium the sound propagation is slower. Usually in solids sound propagates with higher speed with respect to liquids and gases. A solid is surely denser than a liquid (with exception of ice!), so it should propagate sound more slowly. However they are also stiffer, so in practice in solids the effect of the force binding the molecules prevails on density effect.

Elasticity and density may vary in the same medium according to other physical parameters, such as temperature and, for water, pressure and salinity. Such variables influence at the same time both elasticity and density so that if you increase temperature and either pressure or salinity or even both in a medium, the sound speed will increase too.

In the specific case of a metal bar emitting a sound upon being hit there are also other parameters to consider in order to predict the produced sound: the bar shape, dimension and mass, the impact point, the shape and material of the hitting body and boundary conditions (whether the bar is free, clamped or just suspended). The bar once hit is slightly deformed. Owing to the elasticity of the material the mechanical energy stored into the initially deformed shape is transformed into elastic waves, some of them are irradiated into the surrounding air and caught by our ear as sound. Generally speaking the least elastic the bar and the most energy is dissipated as heat causing the sound to be quite similar to a dull thud. In elastic materials the sound has quite a complex spectra made by a wide band noise, due to the impact, to which a set of peaks is added (partial components), each corresponding to a single normal vibration of the bar.

Since the partial components are not in harmonic ratio with each other it's not easy to hear any specific and clear note. However the first partial (called 'fundamental') usually is the one with most energy within the spectra and the perceived pitch most probably will exhibit such a frequency .

By either heating or cooling the bar the thermal expansion will modify all the elastic properties and the density but the thermal coefficient of metals is normally so low that the frequency is little influenced when temperatures varies within the 0-100 °C range.

In Nitinol on the contrary, a characteristic shared with all shape memory alloys, since we may consider density variation as negligible, elasticity strongly depends on phase. When the phase transition starts, elasticity and consequently sound speed largely varies. However the vibration wavelength can't possibly change, since it depends only on the bar length, therefore it is frequency which undergoes a dramatic change.

The reason why elasticity changes so dramatically during phase transition is that it all depends

on how strong is the interaction between neighboring atoms. When Nitinol undergoes a phase change the position of the atoms changes as well and their interactions are greatly influenced. In normal metals instead the atoms position is not completely reorganized in a different geometry owing to the temperature change: in this case the atomic interaction force is such that in the 0-100 ° C range atoms move very little.

4 – Muscle wire: a sensore & actuator

Nitinol wire with a diameter ranging from 25 to 300 micron is also known as *muscle wire*[*1] (one of its commercial labels is Flexinol). It is often employed as electrical actuator in reproducing and mimicking muscle movement in robots. Thanks to its capability to contract when current flows through generating heat by Joule effect, it can generate forces that easily are one thousand times higher than the wire weight.

Lab Goals

- Reflecting on the meaning of "phase" in a material
- Understanding the concepts of sensor and actuator
- Understanding muscle wire mechanism and some of its applications.

What's to be observed

The wire set-up known as "actuator triangle" can be exploited to convert the tiny movement of the contracting wire in a larger vertical movement with a distinct gain over the lift produced by a simply linear set-up.
It is fundamental that a *bias force* is applied to the wire as it cools down since the *one-way* alloys doesn't recover spontaneously the original length. If after a contraction no force is applied elongation will be only partial and in the heating cycle there will be a minimum contraction with a weak resulting force

Materials and equipment (for a single group)

- muscle wire (L = 10 cm ~ , ~100μm ∅)
- electric cable
- battery (3V)
- switch
- graph paper
- wood planck (20 X 30 cm) and support
- plastic envelope + paper clip
- 10 grams masses
- 2 drawing pins
- ruler

Link to the videoguide

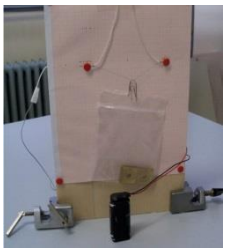
Read the QR code on the right, or link to

Home > Labs > Memory metals > 4 – Muscle wire as actuator> Videoguide

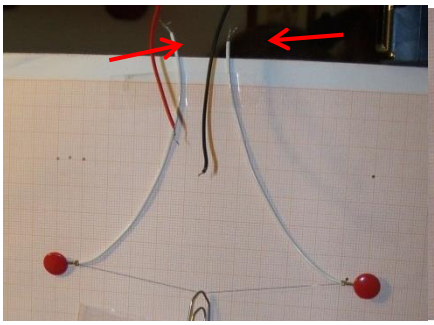
Background reading

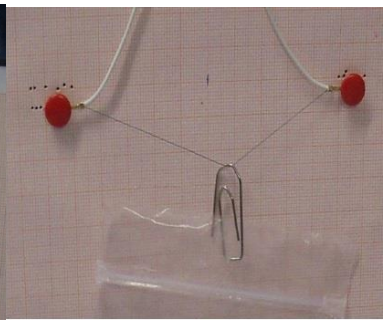
Experimental protocol

A – Setting up the circuit



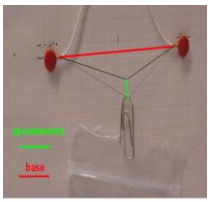
1 – Cut a piece of graph paper and cover the wood plank fixing it with the drawing pins. 2 – With two more drawing pins going through the two tiny terminal rings fix the nitinol wire to the planck (left and centre bottom pictures). 3 – Connect the two electrical wires previously soldiered to the muscle wire with two other long electrical cables – approx. 25 cm each (centre bottom picture- see arrows). Soldier their ends to the battery and insert into the circuit a switch by cutting one of the two cables. 4 – Make a hole in the upper end of the



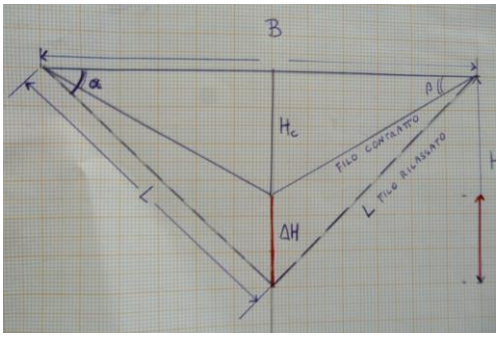


plastic envelope and slip a paper clip into it: it will work as a hanger and you will position the envelope exactly at the center of the Nitinol wire (see right picture). Put the desired masses into the envelope but never exceed a total of 30 grams! You may also use cents coins as suitable masses

B – The actuator triangle



Change the triangle base B del keeping the same applied mass Check that it hangs perfectly at the middle of the wire. You can exploit the grid on the graph paper to reposition the drawing pins each time you change the base length and also to actually measure it.



Measure H, the triangle height when the wire is still "relaxed" (= prior to the passage of the current). Turn on the switch and close the circuit. Be quick to sign with a pencil the height H_c of the new triangle formed by the contracted wire and immediately after that switch the current off or you will overheat and permanently ruin the wire! Under the weight the wire will elongate beck to the original length. Now measure Hc directly on the graph paper and calculate the vertical shift length DH = H - H_c. Is there any special relationship linking B and DH?

DO NOT exceed 3V and switch the current immediately off.

If the current keeps flowing for more than a few seconds you risk to damage the wire permanently..

DO NOT hang a mass heavier than 30-35 g maximum

Connections between the different parameters

B = base *(the distance between the two drawing pin fixing the wire to the wood board)* ***L= length of the triangle side****. The total length of the wire is 2L*

%2L = elongation in % *of the total length. Usually it doesn't reach beyond 3-5% without risking of damaging the wire permanently even if theoretically it may reach 8%. (It is possible to think about the meaning of linear expansion coefficient which exhibits a negative value in Nitinol).*

H = triangle height *when the wire is relaxed (from L and B with Pitagora theorem)*

H_c= triangle height with contracted wire

DH = H-H_c *vertical shift due to the wire contraction*

F_I *= force exerted by the wire in a linear contraction (it is the so called "recommended recovery force"- usually given by the manufacture)*

%F = Sin(Arctan((2H)/B))) *% of F_I exerted to lift the weight. The remaining force acts horizontally on the two bolts (drawing pins)*

F_p= 2*%F* F_I *force exerted by the wire to lift the applied mass*

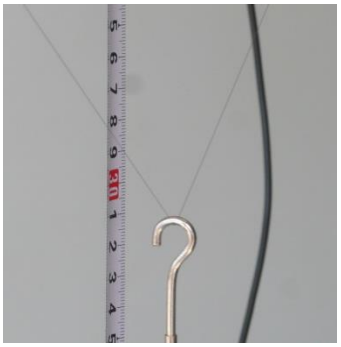
As you keep all the other parameters constant (applied mass, length and type of wire) by varying the base B of the triangle you obtain a corresponding height H. As a consequence the vertical shift produced varies as well due to a change in the pulling force.This can range from 0 to a double valueof the force exerted in the linear contraction (it is a double one since there are two forces pulling along each one of the triangle sides. Starting from the maximum length possible for base ***B*** *and reduce it progressively of 1 mm at a time moving one of the two draw pins and repositioning the hanging weight in the middle.*

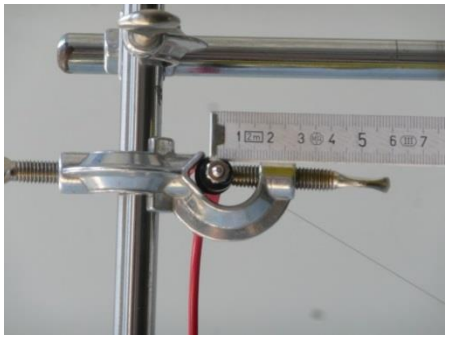
The Flexinol wires, as thin as human hairs and just ten centimetres long, are quite suitable to make students appreciate the possible use of nitinol in robotics. However if you are more interested in the quantitative aspect rather than in the demonstration you may use longer wires (diameter~150μm, length~1 m). It will be much easier with big actuator triangles to obtain the quantitative relationship between the length of the base and the vertical shift of the applied mass produced. The wire is fixed at the two ends to two electronic plugs with their screws and connected to a power pack with the electric cables. You can find examples of data collection at

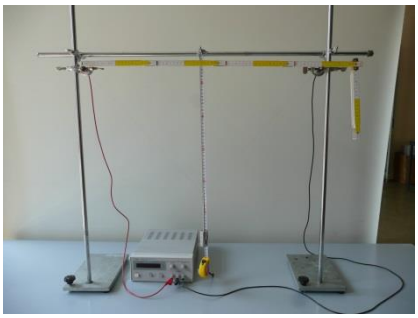
Home > Laboratori > Memory metals > 4 – Muscle wire come attuatore >

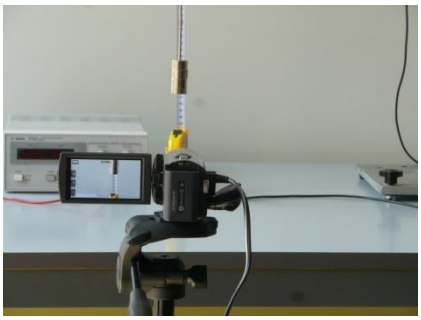
With this set up you can also investigate in detail how the wire behaves when the current flows through appreciating the different speed according to the current intensity. (in this case you should videorecord and then analyze with Tracker- see activity 2 of this module.

The set up: details









For senior students who have done some trigonomethry

Ask the students to draw a scheme of the set up

a) before the current flow

b) during the contraction

with all the acting forces.

With trigonometry they should find suitable relations between the known (triange base, wire length, weigth of the applied mass) and unknown (vertical shift produced; force exerted by the wire, total and percentage elongation) parameters.

C – Nitinol wire diameter

Nitinol from the "relaxed" martensite phase to the contraced austenite one changes its dimensions. You may try to test it.

a)**length**: through the actuator triangle

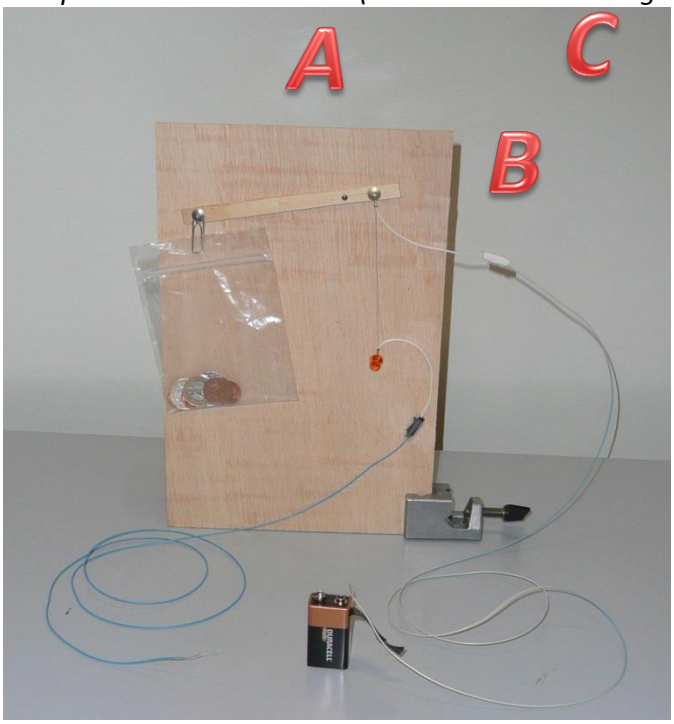
b)**diameter**: you may try to evaluate the change in diameter through a change in the diffraction design (see " measuring the diameter of an human hair" at http://oldphysicscom.unimore.it/index.php?page=laboratori_didattici or just Google "*Measuring the Diameter of a Human Hair by Laser Diffraction.pdf*").

as an alternative you may just take a picture of the two diffraction pictures and then analyze them with Tracker (modality: spectra analysis).

As you have already seen in activity 2 Nitinol during the phase transition changes its resistivity. A Flexinol wire exhibits a very high electrical resistance if compared with other metals such as copper. R counterintuitively decreases as the wire heats up and contracts while usually in metals an increase in temperature actually determines an increase in R too. The decrease in R is partly owed to the shortening legth and the thickening diameter but mostly due to the different crystal lattice structure. It may be interesting to ask students to study and plot the wire IV curve.

With a few more material it is possible to build levers based on nitinol. See the picture in the following page. The distance from the *pivot B to C (connection of the lever with the muscle wire) and A (hanging point for masses) may be expressed through a specific ratio. For instance if the ratio is 5:1 (AB = 10 cm; BC = 2 cm) it means that for each millimeter moved by the lever end linked to the wire, the end with the mass hanging will move 5 mm.*

Building Instructions: *Make a hole in 3 points A,B,C of the lever. Put the two screws at the two extremities: on the right the screw will be slipped inside the terminal ring of the nitinol wire . nail the lever to the point B: leaving the lever free to rotate . Fix the second end of the wire with a drawing pin: the wire should be tense and perpendicular to the lever it is not contracted. Complete the circuit (see actuator triangle) and hang the masses to A*



References

See

Home > Labs > Memory metals > Background reading

And

Lisotti A., De Renzi V., Villa E., Albertini F., Rozzi C.A., Goldoni G.-*Educational pathways through nanoscience: nitinol as a paradigmatic smart material*- Phys. Educ. 48 (2013) 298-311. -

Since on NANOLAB website there's not a background reading specifically targeting sound properties in solids, we present here a list of web resources to research the topic further

- *At* http://fisicaondemusica.unimore.it/Modi_normali_di_una_barra.html *you will find an animation of the bar oscillatory modes , while , for a more in depth analysis see* http://fisicaondemusica.unimore.it/Frequenze_proprie_della_barra.html *, e* http://fisicaondemusica.unimore.it/Equazione_delle_onde_nella_barra.html
- http://www.springerlink.com/content/bw264x51284733t5/ *investigating bones age with ultrasound*
- http://en.wikipedia.org/wiki/Scanning_acoustic_microscope
- *Acoustics of thin Ice http://www.acuvib.com/ice.html*

Credits

The last activity has been partially adapted from the NANO-CEMMS activity on Nitinol https://nano-cemms.illinois.edu/materials/nitinol_basic_lever_desc of the University of Illinois in Urbana Champaign USA

Finding materials and equipment

Nitinol wire can be purchased at

- www.mindsetsonline.co.uk 1 m approx 7,14 pounds (without delivery)
- SAES Getters Group - Lainate Milano - Italia
- In USA there are many retailers , one of them is http://www.imagesco.com/ see "nitinol products".

Nitinol springs can be purchased at

- www.mindsetsonline.co.uk UK type "smart niti spring" or "PAC SW3 " in the quick

search slot on the left. Cost £5,40 each, (without delivery). Payment in advance and in pounds.

- www.futurashop.it Futuraelettonica- Italy - under "nuove tecnologie" (in the list on the left column). Cost 16 € (without delivery).
- SAES Getters Group - Lainate Milano - Italia
- Imagesco http://www.imagesco.com/ see "nitinol products". USA
- In Germany an industrial manifacturer of strips, wires, pipes and sheets http://memry.com/

In addition to Niti springs you need:

a) The **double hole plaquettes** to hang the springs to the lab stand and the mass hanger to the spring respectively**.** You can buy them at www.mindsetsonline.co.uk . Type "Wire Holder plates" or the corresponding code [211-011] in the quick search slot. Cost £ 2,16 (10pz) delivery costs not included.

Alternatively you can make home-made ones by drilling stiff plastic slates or even already punched metal slates like "meccano" (at the hardware 0,30 € each): If you use metal ones carefully check that they are isolated from the rest of the circuit.

b) The **plugs** are sold in any electronic material shop. Cost: 0,80 € each;

c) **Thermoshrinking plastic tube** is sold in electrical or electronic materials shops.

The N**itinol bars** used in our experiment were manifactured by IENI CNR, Lecco (IT). SAES Getters Group sells bars but of larger diameter.

- The control bar can be manifactured by a blacksmith or probably found in an hardware store. We used an iron bar but inox steel or different metals are ok. The really important thing is that its dimensions are similar to the Nitinol bar.
- The microphone that we have actually used has a USB plug and audio board incorporated GO Mic by Samson. Cost 39 €; in musical instruments shops .

Software

- Tracker - Video analysis software. Free download at http://www.cabrillo.edu/~dbrown/tracker/ - It belongs to the Open Source Physics repository.
- Audacity - Software for audio recording, editing and analysis. Free download at http://audacity.sourceforge.net/

Android apps

- Pure Audio Recorder http://www.appbrain.com/app/pure-audio-recorder-free/jp.gr.java_conf.nand.pure_audio_recorder_free
- SpecPro Analyzer 5,99 euro

- SpecScope Spectrum Analyzer 0,99 euro.