

Teaching the behavior of superalloys at high temperature using model metals for easier test conditions: Use of pure lead to visualize tensile creep deformation

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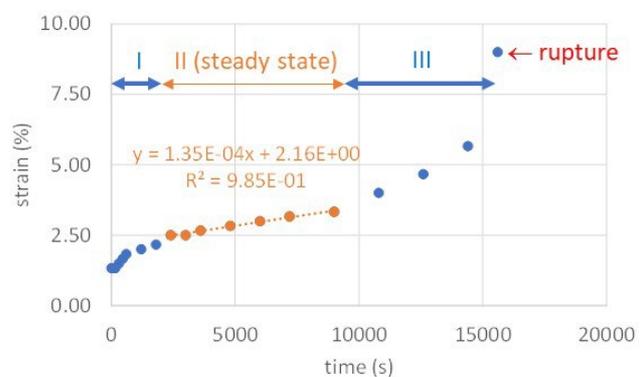
Abstract: Creep deformation is an important phenomenon leading to failure for high-temperature working components present in aeroengines, combustion turbines or industrial shaping tools. When it has occurred in traction, it is composed of three successive steps. It can be useful, for students in Master of Materials or Metallurgy, to practically observe these three steps of progression of creep deformation. Investigating the dependence of the steady state deformation on temperature and on the applied stress is also of high practical interest. Unfortunately, very high temperature tests imply the use of expensive experimental means as well as severe safety conditions to respect. In this article the illustration of the high temperature creep of highly alloyed steels and superalloys is experimented using lead and a simple and cheap material. The test bench to use can be also very simple and it is even possible to assemble with items that everybody can find at home! It is demonstrated that lead, room temperature, centimetric samples easy to prepare, and several kilograms hung are enough to clearly observe the three stages of creep as well as the final rupture, and this in the classical duration of practical works (e.g. 4 hours) thanks to a wise choice of the stress for a given temperature. Furthermore, specifying the steady state creep rate for several (temperature, stress) couples allows estimating the values of the constants present in the laws of dependence of the secondary creep rate on both stress and temperature. This helps to better understand the Larson-Miller method.

Keywords:

mechanical practical works,
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lead as model alloy,
tests at room temperature,
law's exponent,
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1 Introduction

Mechanics of materials are a broad domain of materials science, beside thermodynamics of materials, microstructures formation, microstructure stability at high temperature, corrosion... Rigidity, elasticity, plasticity, strength, hardness, toughness, rupture, fatigue... depend on the type of cohesive forces, the chemical compositions, the crystalline network, the alloy's grain size ... They also depend on the conditions of solicitations (Baïlon & Dorlot, 2000), particularly on temperature.

Indeed, temperature generally influences the mechanical behaviors of materials. When it is low for the considered material this one can be fragile and its toughness is low. When temperature becomes higher than the ductile brittle transition temperature (DBTT) of the material, this one can demonstrate ductility and higher toughness, as well as a greater reproducibility of strength. When temperature becomes very high, viscous plasticity appears and can lead to rupture after a more or less long duration, even if stress and temperature stay constant. This phenomenon is called “creep”.

The metallic materials are particularly concerned by this behavior when they are mechanically stressed at temperature close enough to their solidus temperature or melting start temperature (Sims & Hagel, 1972). In order to resist against creep deformation and rupture, one must use particular materials in the hottest parts (combustion chambers, vanes, blades, disks...) of in aeroengines or in power generation combustion turbines, or in any industrial components working too at high temperature under mechanical solicitations. These particular materials are metallic alloys especially designed, notably at the scale of their microstructures (Bradley, 1988). Based on nickel or cobalt – elements presenting high melting points ($> 1450^{\circ}\text{C}$) – and benefiting from coarse grains (or single grain structure), strengthening by heavy atoms in solid solution or by various micrometric or nanometric precipitates (intermetallics, carbides, oxides...), these high performance alloys – called “superalloys” – are more and more used (Donachie & Donachie, 2002) in transportation, energy production or heavy industries. This explain why superalloys are present in the teaching programs of students preparing a Master in Metallurgy or in Materials Science, in the same way as cast iron, steels or aluminum alloys. Beside theoretical courses and exercise sessions or tutorials, practical works can be of high usefulness for a better learning.

Exploring the mechanical properties at ambient temperature of ferrous or non-ferrous alloys is classical and does not require very expensive apparatus. For instance, not too expensive indentation machines or traction machines can be present in practical works rooms. In contrast, investigating the high temperature mechanical behaviors of

superalloys is not possible everywhere, due to the high cost of purchase and maintenance. Nevertheless, in some cases, some phenomena can be reproduced at lower cost with a minimum of materials, with almost the same explanatory power. This is in this frame that one will present here cheap and not dangerous (e.g. no risk of burn) test alloy and little equipment able to allow students observing the creep phenomenon, and even starting to exploit results to estimate some important parameters involved in the laws describing the influence of stress and of temperature on the deformation kinetic.

2 A few basic concepts about creep

Creep is thus a phenomenon occurring when an alloy is stressed when its temperature is lower than – but not far from – its melting start temperature. A simple temperature criterion to respect for observing this viscous plastic deformation is:

$$T_{\text{alloy}} \text{ (K)} > f_{\text{cs}} \times T_{\text{ms}} \text{ (K)}$$

Where T_{alloy} is the absolute temperature (supposed homogeneous, expressed in Kelvin) of the alloy when it is subjected to the stress, f_{cs} is a factor depending on the alloy's composition and microstructure (often taken equal to 0.5) and T_{ms} is the temperature of the melting start of the alloy. For instance, the creep deformation of a stressed superalloy which starts melting at 1250°C ($\cong 1523$ K) should be detectable at $\cong 760$ K, i.e. $\cong 490^{\circ}\text{C}$. In fact, due to the solid solution hardening and/or to the strengthening particles that its microstructure contains, a better value to choose for f_{cs} can be 0.7 and thus superalloy is rather expected to know creep for temperatures higher than 1070 K (i.e. 800°C).

Generally creep is progressing in conditions of varying stress and varying temperature. The best way to evidence it is to heat a sample at a high temperature, to maintain constant this temperature, to apply progressively a stress and to maintain this one constant on long time. The creep phenomenon that one can observe in these conditions can be depicted by the scheme given in Figure 1, on the right side of which is also shown a superalloy sample after creep-rupture test. One can see that not only one, but several cracks were propagating before the main one finally caused the rupture.

The creep deformation does not progress at a constant deformation rate. Indeed, after a fast but decelerating deformation (primary creep stage) the deformation rate becomes stabilized. Thereafter, during a long time, the alloy deforms at a low constant rate (secondary creep stage, also called “steady state”). Except in cases such as logarithmic creep (which may occurs when both stress and temperature are low), the low deformation

finishes by re-accelerate, until the final rupture occurs. The kinetics and durations of the three creep stages depend on the applied stress and on the temperature. When increasing separately or simultaneously, both parameters accelerate deformation and shorten the three creep stages. They decrease lifetime and, in many cases, increase the total permanent elongation. This is illustrated in Figure 2, in the right side of which several laws of dependence of the steady state deformation rate on temperature and on stress are mentioned.

In most cases the creep deformation is of a viscous plastic nature, which means that the deformation after interrupted creep remains permanent. This dimensional evolution, in the case of tensile creep, can be simulated by a mechanical model (Figure 3, left hand) involving two springs (stiffness constants noted k_1 and k_2) and two viscous pistons (viscosities noted η_1 and η_2).

Figure 1. Scheme depicting the progressive deformation of a metallic sample submitted to a constant tensile stress and at a constant homogeneous temperature; on the right: macrograph of a superalloy sample finally broken after about 1000 hours at 900°C under an applied stress of about 50 MPa.

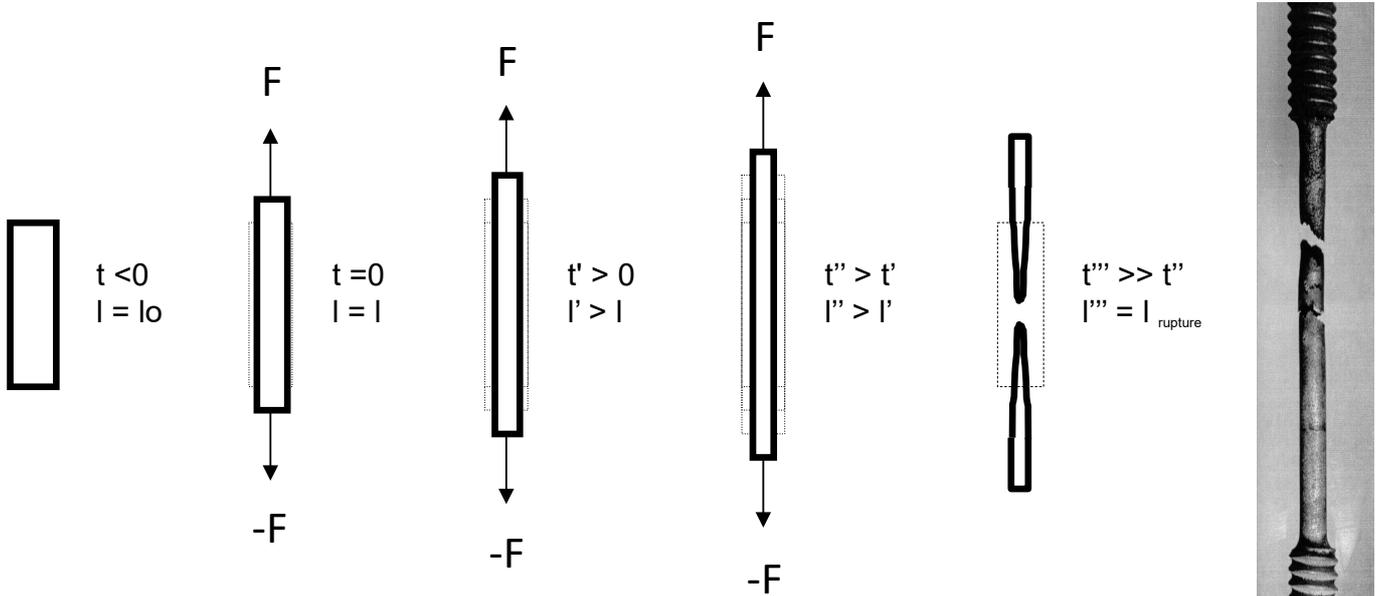
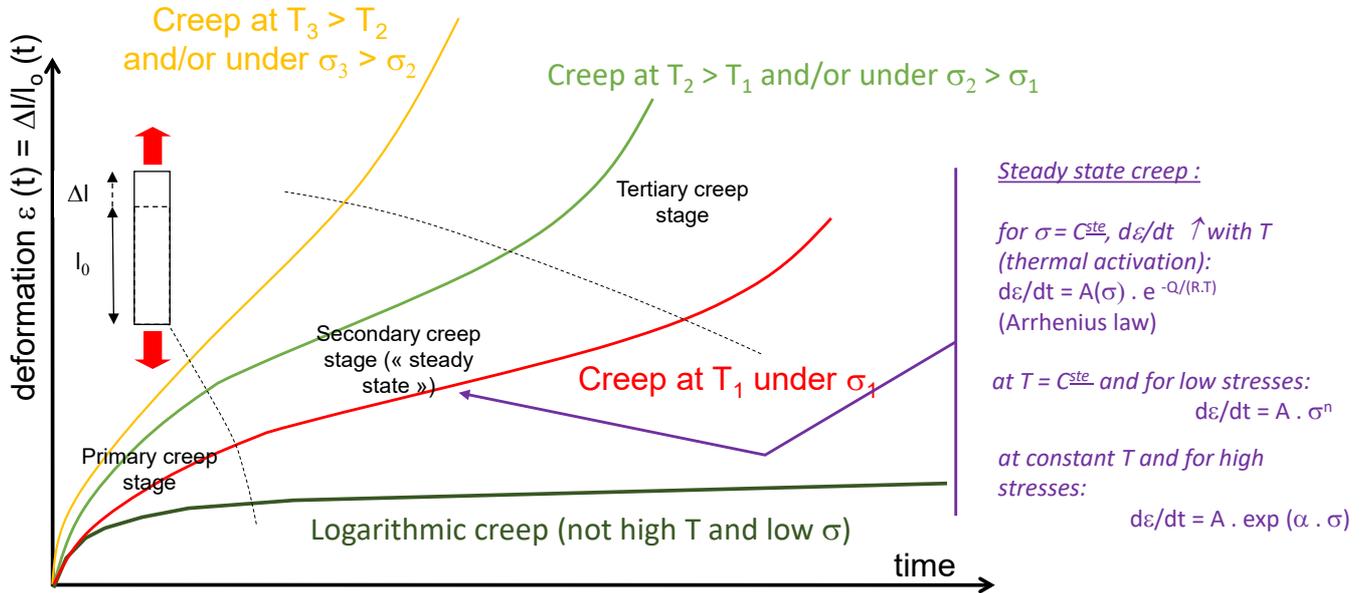
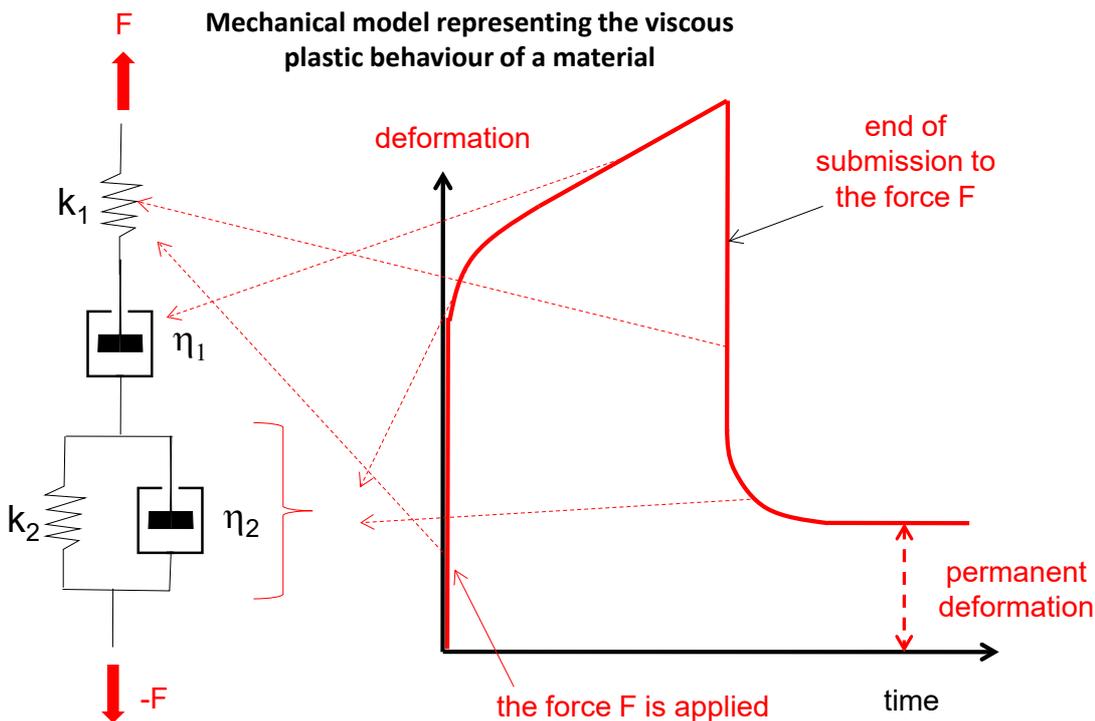


Figure 2. Illustration of the three successive stages of the creep deformation progression in the case of isothermal and constant tensile loading; qualitative graphical description of the dependence of each stage on temperature and stress; three formulas quantitatively linking the steady state deformation rate to either temperature or stress.



The behavior of this model when rather rapidly loaded then maintained subjected to a constant force, and thereafter freed by the rapid suppression of this force, is described on the right side where the existence of a permanent deformation is evidenced.

Figure 3. Mechanical model simulating the creep deformation (viscous plastic model) and its qualitative dimensional evolution if a constant force is applied then removed.



There are two main types of creep: the “dislocation creep” and the “diffusion creep”. The later one occurs at very high temperature, close to the solidus temperature of the alloy. It is due to atomic diffusion in the direction of the applied tensile stress, intragranular (in volume) and/or along the grain boundaries. The former one is more frequently encountered. It is promoted by the movement of the linear (one dimension) crystalline defects called dislocations. The ones are complex curves lines which are not easy to represent. This is why one generally starts by speaking about “edge dislocations” and “screw dislocations” (Figure 4), which are geometrically far from the real dislocations but which facilitate explaining how dislocations may definitively deform the crystal, and thus the alloy itself if it is single-crystalline, or a grain in a polycrystalline alloy (Figure 5).

During “dislocations creep”, the dislocations initially present in the single-crystalline alloy or polycrystalline alloy, move under the effect of local shear stresses, rather easily due to the thermal agitation. They meet obstacles which can block them but also which may favor their multiplication by acting as Frank-Read sources (primary creep stage). A great number of dislocations in a same location finishes by creating cavities and causing the secondary-to-tertiary creep stage transition, and finally cracks propagation, leading to the rupture by stress concentration. Pure metal or simple alloys know fast creep deformation. In contrast, superalloys resist creep since heavy atoms in solid solution and finely dispersed hard precipitates of various types (intermetallics, carbides...) obstruct efficiently the dislocation sliding.

Figure 4. Two simple but pedagogically important models of dislocations: the “edge dislocations” and “screw dislocations”, here in a simple cubic crystalline network (CsCl-type).

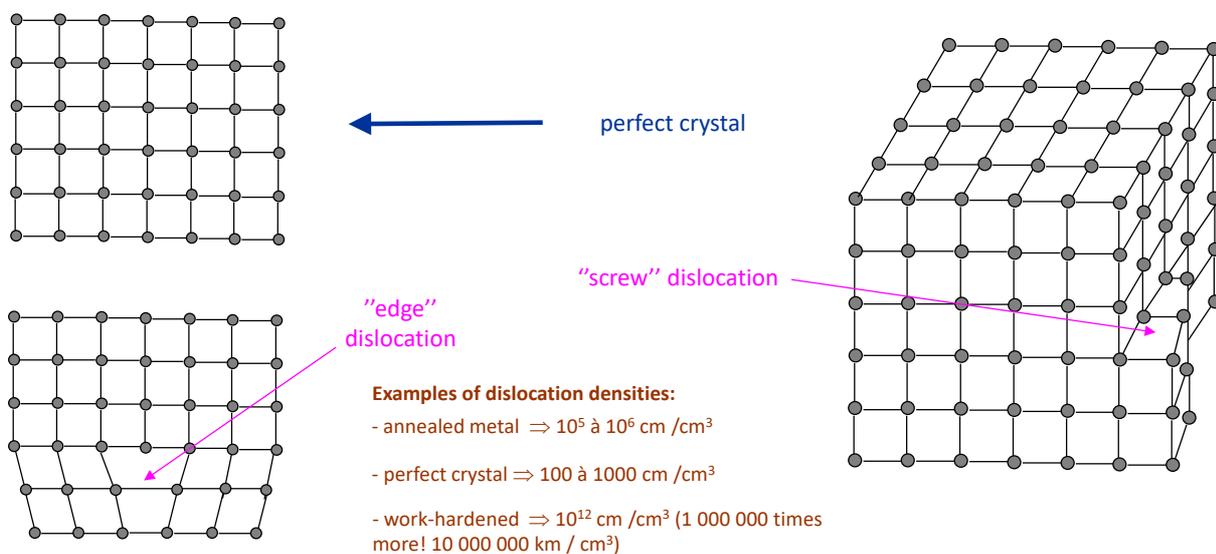
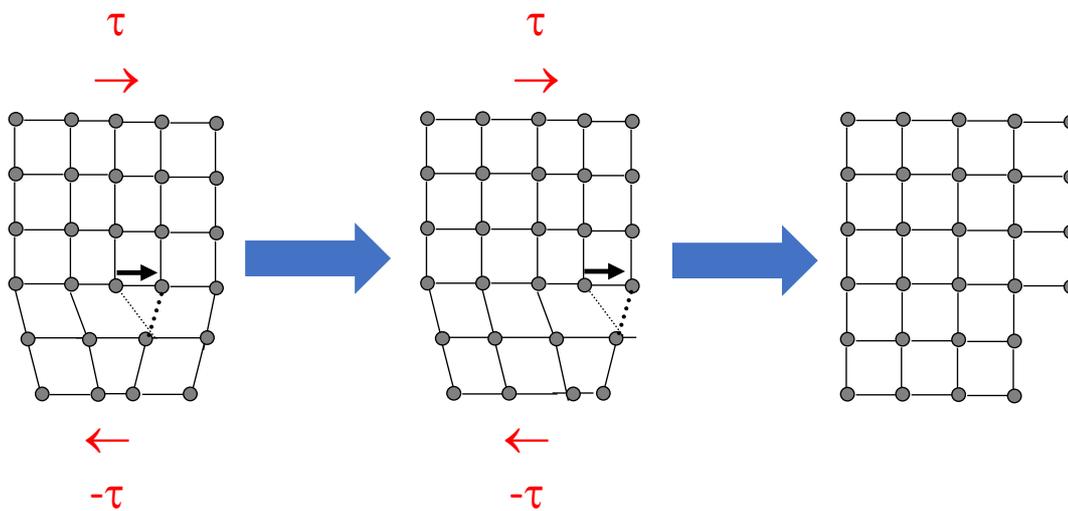


Figure 5. How a local shear stresses couple (τ and $-\tau$) may induce the displacement of an “edge dislocation” in a simple cubic crystalline network, until causing a sliding step in crystal periphery (elemental “brick” of the permanent deformation).



3 Materials and methods

3.1 Choice of a suitable metal

To allow students observing the creep phenomenon at ambient temperature one has to choose a material which:

- Is metallic and polycrystalline to allow viscous plastic deformation by dislocations sliding
- Has a melting start temperature low enough to allow observable creep
- Is easy to cut by students (e.g. using simple scissors)
- Is not too expensive

Several metals respect more or less these criteria: tin, lead ... The melting point of tin is 232°C , as is to say 505K . For $f_{\text{cs}} = 0.5$, the test temperature must be higher than 253K , i.e. -21°C . The melting point of lead is 327°C , as is to say 600K . For $f_{\text{cs}} = 0.5$, the test temperature must be higher than 300K , i.e. $+27^{\circ}\text{C}$. If the rule valid for superalloys also applies for these two low temperature melting metals, tin is a better choice than lead. Unfortunately tin was not present by us with pieces with size great enough while lead was available as rolled coil with a width of more than 10cm and a length of more than 2 meters. It was thus possible to prepare tensile samples of lead long enough to hope measuring elongations with an acceptable accuracy. Lead was then chosen.

3.2 Specimens preparation and choice of the load

A series of tensile samples were cut with domestic scissors, by targeting a gauge length equal to 150mm, heads with 15mm (width) and several cm (length) as dimensions. Shoulders were designed for progressive transition from the 15mm of the heads width and the 5, 4 and 3mm for the gauge width, to inhibit stress concentration at these junctions. In a first time, most of students will certainly prepare specimen with abrupt section transitions between head and gauge (with $\pm 90^\circ$ angles): in such case premature rupture at one of the two head-gauge junctions will give the opportunity to the practical works supervisor to remind them the problem of stress concentration and the necessity of a much smoother transition.

Photographs of the three cut specimens for tensile tests can be seen in Figure 6.

Figure 6. Specimens obtained by simple cutting using classical scissors (before the straight correction of the gauge parts).



The choice of the load to apply was done according to the necessity to produce a stress high enough to promote rather fast creep but not too high to prevent any rupture during loading. In a recent work (Berthod, 2025) a tensile test was carried out using an universal testing machine (UTM) available in the practical works room of the “Institut Universitaire de Technologie” Nancy-Brabois (University of Lorraine). It was performed, at a constant low strain rate, on a traction specimen with a large gauge to allow the force cell equipping the UTM (capacity 100kN!) being strong enough to be measurable with an acceptable accuracy with the 100kN cell (capacity unfortunately much too high). During this earlier test one obtained a strain-stress curve showing a yield stress (YS) of about 8 – 9 MPa

(threshold to exceed) and an ultimate tensile stress (UTS) of about 14 MPa (to do not exceed). These results, which were obtained for a room temperature (about 18-20°C) were considered as rather consistent with those of Muramatsu *et al.* (2014) who found the same level of values (however slightly lower). In the present case, despite that the temperatures, at which the tests which will be performed, will be lower (tests run outside, during winter), one can think that the YS and UTS would be higher than the values obtained at room temperature. Nevertheless, because of the impossibility to perform outside tests with a UTM, one kept this YS and UTS values for the load determination.

So, taking into account that the thickness of the lead coil is 1.25mm, for width of the gauge part of the creep samples comprised between 3 and 5mm, the constant applied force will be comprised between 33.8N and 52.5N for a gauge width of 3mm, to between 56.25N and 87.5N for a gauge width of 5mm. This corresponds to a mass to hang comprised between 3.4 and 5.4 kg for a gauge width of 3mm and between 5.7 and 8.9 kg for a gauge width of 5mm. In order to perform all tests with a single hanged mass, 5.4 kg was selected. Since the width was not really very constant all along the gauge part, each specimen was subjected to a series of width measurements followed by the calculation of the average width (Figure 7, left hand). It was considered that this was acceptable for the representativity of the monitored progressive elongation of the gauge part. Of course rupture is each time expected to occur in the less wide location in the gauge part; consequently the lifetime (if the test is not voluntarily stopped before for any reasons) will be taken with caution.

3.3 Test bench

To reach the selected mass to hang with simple accessible items, there are numerous possibilities: bottles or container filled with water, usual metallic parts for using Roberval balance, bag filled with stones ... To experiment creep on the lead tensile specimen one used three small dumbbell discs ($2 \times 2\text{kg} + 1 \times 1\text{kg}$) of halters destined to weightlifter amateurs. Added with a plastic-coated steel wire and a small clamping device to tighten on the bottom head of the tensile specimens, one obtained 5.4kg (Figure 7, middle, bottom). Temperature was controlled with a simple domestic thermometer (Figure 7, middle, top). An amateur's device can be conceived and realized by the students themselves in few minutes. Here several particularly rudimentary devices were used (an example in Figure 7, right hand) to easily follow the progression of the gauge part elongation ("accuracy" estimated at ± 0.25 mm at best!). Taking into account the lack of

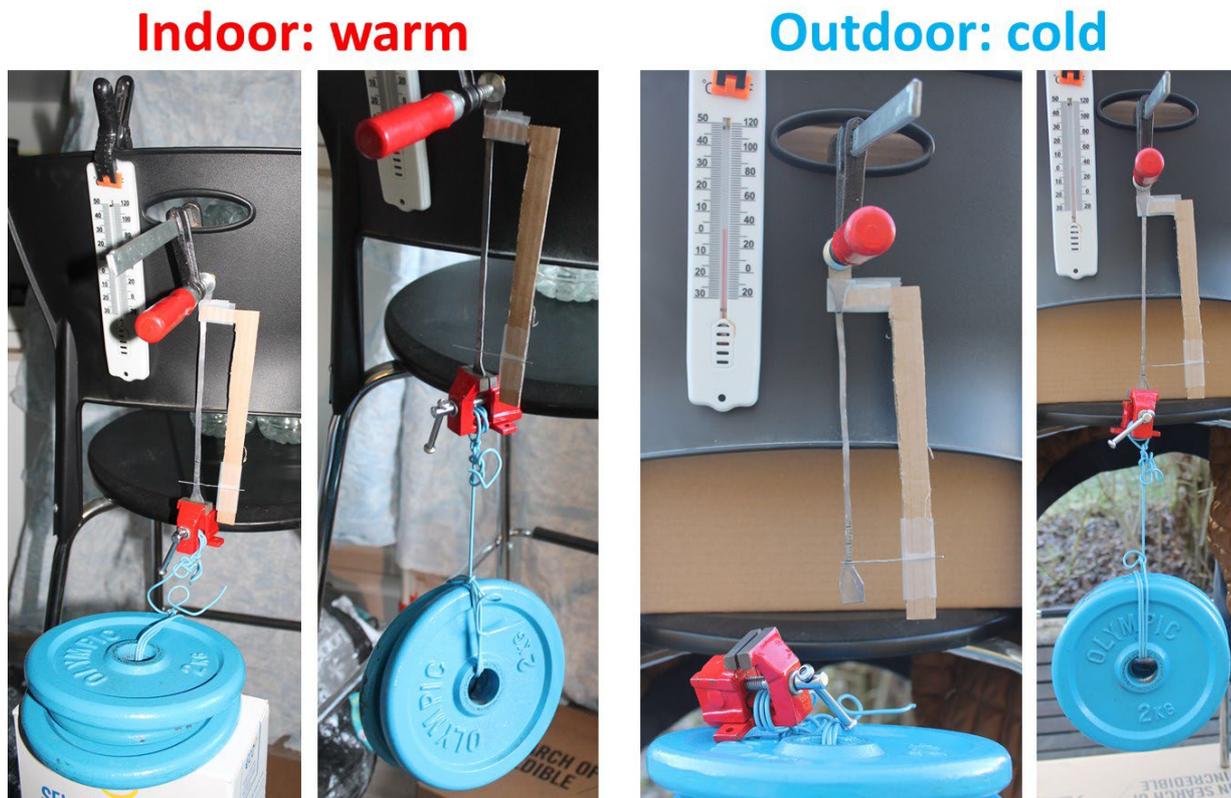
precision of the slightly warped specimens (cutting a 1.25mm–thick lead roll or coil with precision is quite not easy with scissors...) and of the rudimentary “homemade dimensional comparator” is a good opportunity for students to perform uncertainty calculations for the results.

The used test bench (Figure 8) , rather rudimentary, can be used everywhere, indoor (for warm test conditions) or outdoor (for cold test conditions in winter, or hot conditions during a heat wave in summer). Creep tests on three specimens with the three gauge widths 3, 4 and 5mm (thus three applied stresses for the single load value 5.4kg i.e. 53N) were performed outside at a temperature slightly varying temperature between 2°C and 4°C. In all cases, the elongation was noted every 2’30” during the first ten minutes, every 5 or 10’ during the following hour, and every 20 or 30’ thereafter. The choice of time step was done following the observed steady state creep progression, i.e. more frequently for fast creep (high stress).

Figure 7. Some technical details for the successive steps of preparation of the tests.



Figure 8. Rudimentary homemade test bench which can be placed indoor (warm conditions) or outdoor (cold conditions if winter, or hot condition in summer during a heat wave).



4 Results

During the progressive loading, the elastic or elastic + plastic initial strain of the gauge was between 1 and 8mm, depending on the temperature and on the stress.

The deformation was generally fast during the first minutes but the deformation rate decelerated to become rather constant. After a more or less long steady state creep, deformation suddenly accelerated and the specimen failed. The three broken specimen are shown in Figure 9. Each of them is accompanied with its average gauge width and the resulting average stress.

Deformation versus time is plotted in Figure 10 for the three specimen. To facilitate the comparison with previous creep tests performed at 19°C (Berthod, 2025), crept during indoor tests (+19°C), the graph with the same curves is placed beside the corresponding (+19°C) graph for which the axis scales were rated to fit the ones of the present (+2 to +3°C) graph.

Even if observing the tertiary stage of creep was not always possible (due to the periodicity of measurement) to evidence these too fast final deformations, the creep

progression was well composed, after the elastic then plastic deformation during the loading, of the three classical stages: primary, secondary (steady state) and tertiary.

Figure 9. The three crept specimen; rupture zones shown by white triangles.

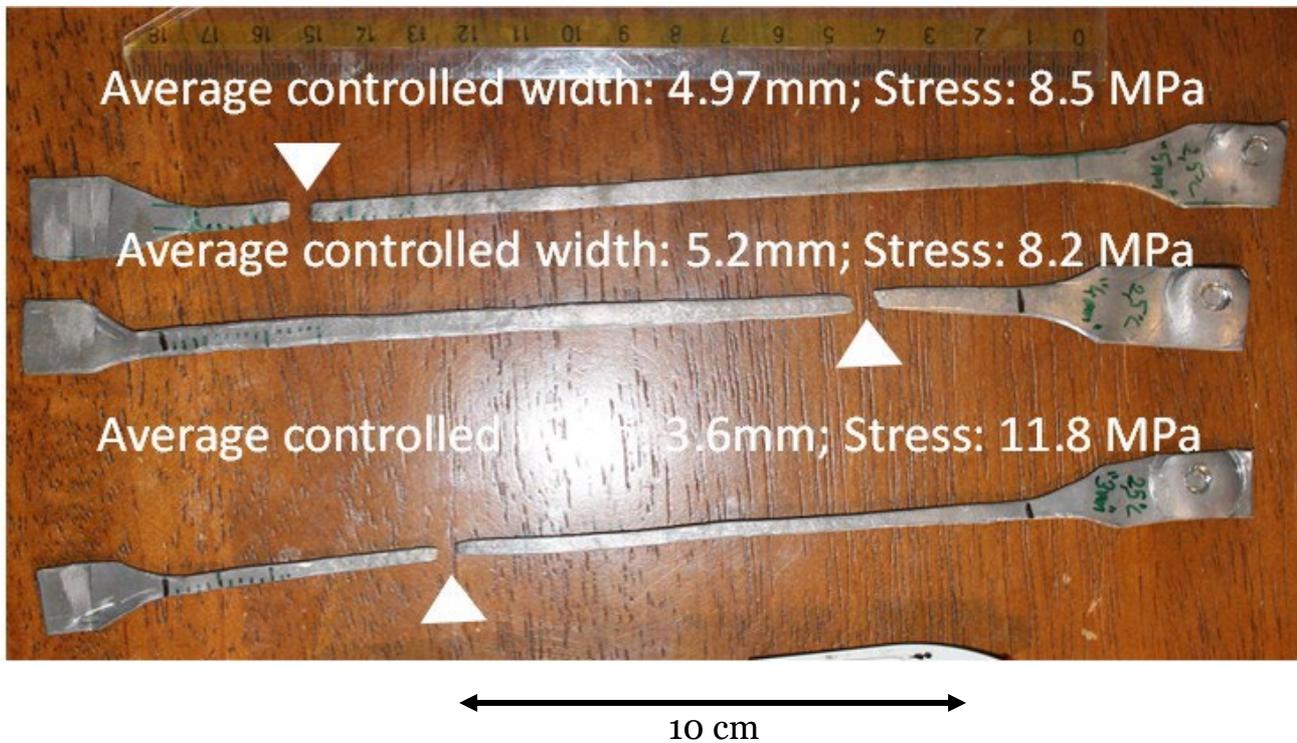


Figure 10. The three deformation curves obtained at $3\pm 1^\circ\text{C}$ (outdoor).

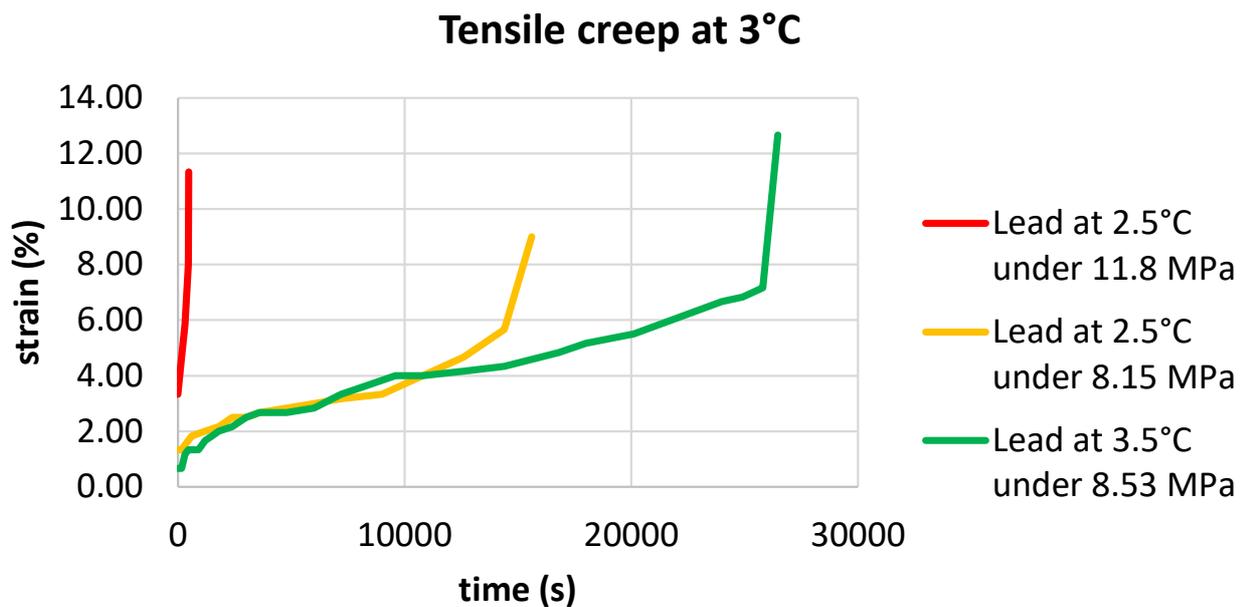
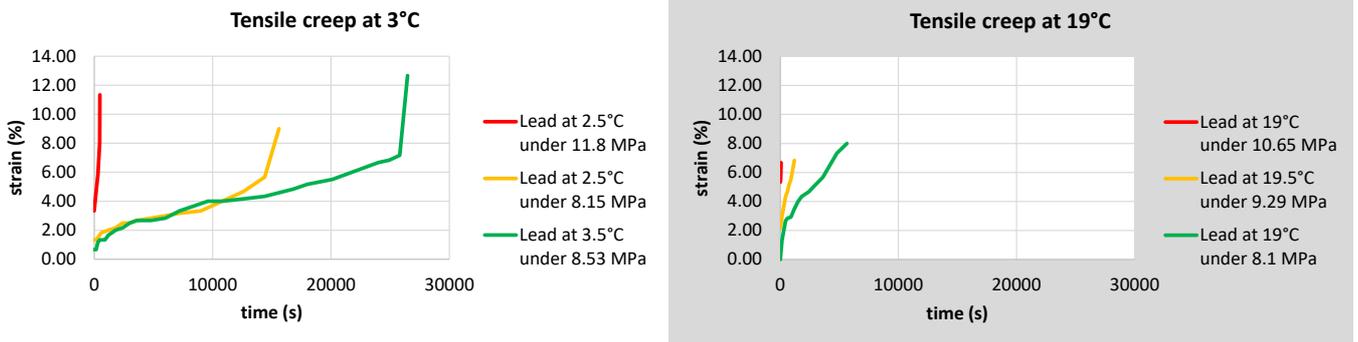


Figure 11. Graphs with homogenized scales for easy comparison between 3°C (this work) and 19°C (Berthod, 2025).

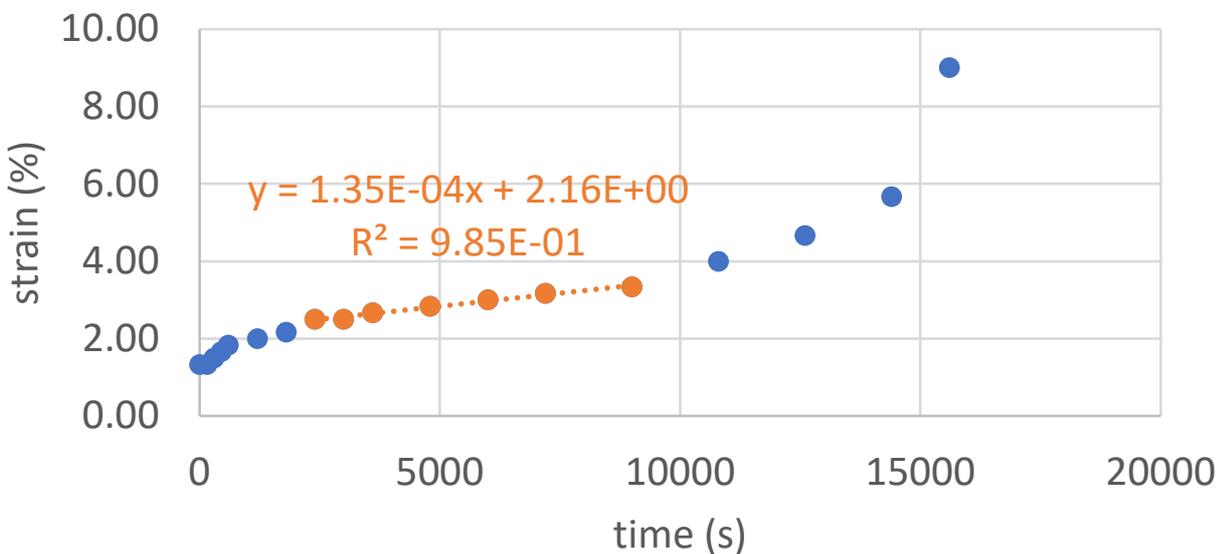


For each temperature the global deformation kinetic increases with the average stress known by the gauge part of the specimen. The higher the temperature, the faster the deformation: this can be easily observed using Figure 11 where the horizontal and vertical scales are homogenized. One can also see that, if the strain at rupture seems to be not dependent on the average stress, it seems in contrast that the strains at rupture are higher for the lowest temperature (what is rather counterintuitive...).

For each deformation curve, the linear part corresponding to the secondary creep stage was isolated and its slope determined for obtaining the steady state creep rate (e.g. $(d\varepsilon/dt)_{II} = 1.35 \times 10^{-4} \% s^{-1}$ in Figure 12).

Figure 12. Example of determination of the deformation rate during the steady state creep.

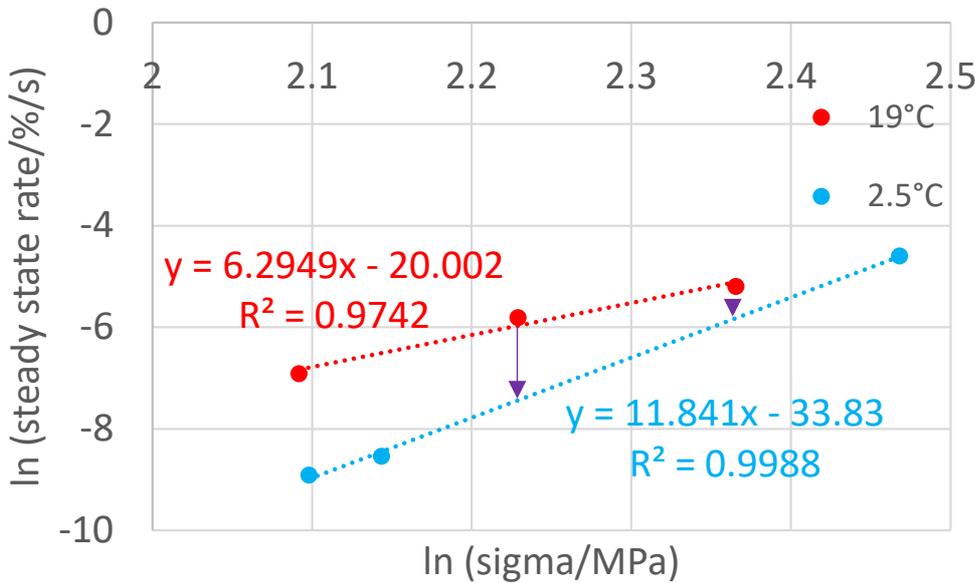
**Steady state creep rate for 8.2MPa at 2.5°C:
0.000135 %/s**



The three obtained values are plotted in a bi-logarithmic graph (Figure 13), where the values previously obtained at 19°C are also added for comparison. For each temperature the three points are rather well aligned; from the equation of the straight line that they define one can specify the values of the multiplying constant A(T) and of the n(T) exponent – T being the test temperature – in the following law describing the dependence of the steady state deformation rate on the constant stress σ:

$$\left(\frac{d\varepsilon}{dt}\right)_{II} = A(T) \times \sigma^{n(T)}$$

Figure 13. Example of determination of the deformation rate during the steady state creep: the present results for 2 to 3°C (this study) in blue and previous results at 19°C (Berthod, 2025) in red.



For instance it seems that n(19°C) ≅ 6.3, which is an exponent value that one can encounter for the “dislocation creep” of superalloys (between 3 and 8). In contrast, the n(2.5°C) ≅ 12 value, too high, is outside this range.

Another important parameter for creep can be also determined: the activation energy Q involved in the Arrhenius law, reminded here:

$$\left(\frac{d\varepsilon}{dt}\right)_{II} = B(\sigma) \times e^{\frac{-Q(\sigma)}{R \times T(K)}}$$

Where R is the constant of the state law of the perfect gases (8.314 J mol⁻¹ K⁻¹) and T(K) the absolute test temperature (expressed in Kelvin).

In fact, it is usual to carefully verify whether the considered phenomenon (here steady state creep) possibly obeys an Arrhenius law, by plotting in a bi-logarithmic graph, the values obtained for at least three temperatures far enough from one another, and to check whether the three (or more) points are sufficiently aligned along a straight line in this type of graphical representation. Here it was difficult to perform tests at three temperatures different enough from one another and one did calculations in the frame of the Arrhenius hypothesis.

By writing two times the equation above for the lowest stresses (8.1 and 8.15 MPa, which are very close to one another) one deduced a value for the (hypothetic) activation energy, according to the following equation:

$$Q = R \times \ln \left(\frac{\varepsilon'_{II}(19^{\circ}\text{C})}{\varepsilon'_{II}(2.5^{\circ}\text{C})} \right) \times \left(\frac{(19 + 273.15) \times (2.5 + 273.15)}{(19 + 273.15) - (2.5 + 273.15)} \right) K$$

With $R = 8.314 \text{ J mol}^{-1} \text{ K}^{-1}$.

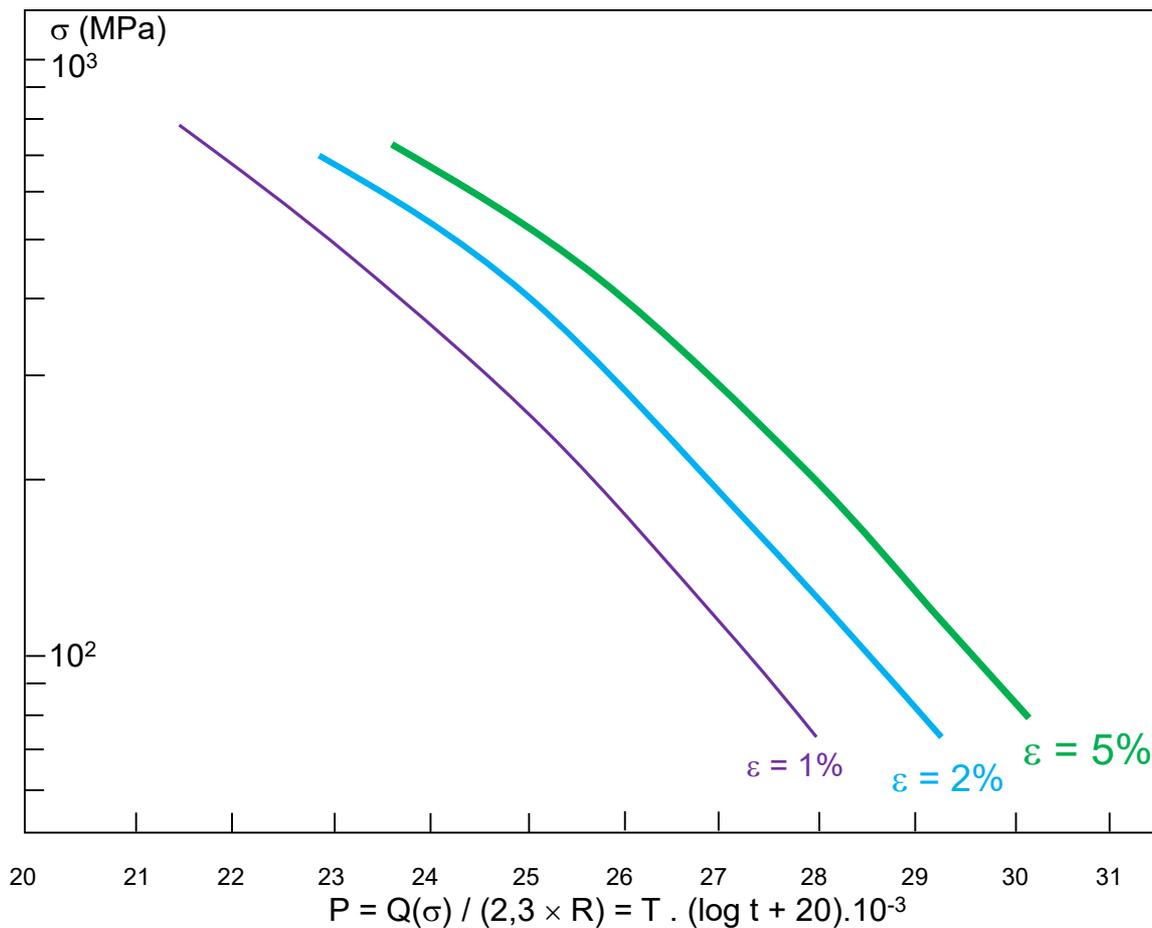
This led to $Q(8.1-8.15\text{MPa}) \cong 200\text{kJ mol}^{-1}$, i.e. a not surprising value since of the same order of magnitude for creep and many of the other phenomena thermally activated.

One can do the same for two higher values of stress, despite that the other stresses are quite different between 19°C and 2.5°C . Indeed, knowing the equations of the straight lines established in Figure 13 one can use them to predict the steady state creep rates at 2.5°C from the values of $\ln(\sigma/\text{MPa}) = \ln(9.29)$ and $\ln(10.65)$. This leads to the following theoretic values of creep rate at 2.5°C :

$$5.8 \times 10^{-4} \% \text{ s}^{-1} \text{ for } 9.29\text{MPa} \text{ and } 3.0 \times 10^{-3} \% \text{ s}^{-1} \text{ for } 10.65\text{MPa}.$$

Using these theoretic values of steady state creep rate at 2.5°C for tests which were not veritably performed, it is possible – again by assuming that the Arrhenius law is respected – to estimate the activation energy for these two other stresses. This gives: $Q(9.29\text{MPa}) \cong 164\text{kJ mol}^{-1}$ and $Q(10.7\text{MPa}) \cong 63\text{kJ mol}^{-1}$. It is known, in the frame of the Larson–Miller method (illustrated in Figure 14), that the parameter $P_{LM}(\sigma)$ value – for a given event (e.g. $\varepsilon = 5\%$) – increases when σ decreases. Thus, the activation energy, which is present in the expression of the Larson–Miller parameter since $P_{LM}(\sigma) = Q(\sigma)/(2.3 \times R)$ with $R=8.314 \text{ J mol}^{-1} \text{ K}^{-1}$, should also increasing when σ decreases. The evolution of Q versus σ observed here, $200 \rightarrow 164 \rightarrow 63 \text{ kJ mol}^{-1}$, for $\sigma = 8.1 \rightarrow 9.3 \rightarrow 10.7\text{MPa}$, is thus qualitatively consistent with what is observed usually.

Figure 14. Example of Larson–Miller abacuses for a superalloy



4 Discussion and conclusions

Lead is already known as an interesting model to simulate the behaviors of other metals and alloys, for instance in association with tin to build experimentally the eutectic-containing binary Pb-Sn diagram (similar to a series of binary systems at higher temperatures, e.g. Fe-C: cast irons) or to evidence the “overcooling” phenomenon (so important for gray cast iron and the graphite–cementite competition) by simple thermal analysis at moderate temperatures. Seemingly it is also a powerful illustrative means of replicating, at room temperature, the high temperature creep phenomenon which limits the lifetime of components made of superalloys to resist mechanical stresses (and chemical aggressions) in service (e.g. inadmissible deformation and sometimes failure of turbine blades).

With cheap materials and simple apparatus, the famous three stages of tensile creep can be reproduced and observed at room temperature (i.e. no need of heating sources), showing the same curve shapes as the superalloys when crept at 1000°C. Furthermore, at 20°C as well as low (and high, possibly) temperatures close enough to 20°C, and for the

same level of stress as in the present study, the steady state creep rate obviously obeys to similar laws versus stress and temperature as the ones known for superalloys. So, besides the possible self-realization of the test bench (and its use) by the students themselves, such tests on lead allow students exploring themselves the steady state rate dependence on stress and on temperature, deducing the parameters featuring in these laws (n , Q ...) and comparing results to what is known for superalloys.

Due to the possible extreme simplicity – and even the rudimentary character – of preparing, performing and exploiting of creep tests on lead, for a limited total cost, a series of bench tests can be put up in order to launch several creep tests (with the same σ and T values for a reproducibility study, or various (σ , T) couples of values) simultaneously to save time. For well chosen values of σ (for a given T), the total creep deformation until rupture can be realized over 3 hours or less to allow carrying out the whole tests and their exploitation.

So, with limited spending, lead allows exploring extensively – at almost ambient temperature – the creep phenomenon and its dependence on the stress and temperature key parameters, this from the specimen preparation to the exploitation of the deformation kinetic, in the frame of a 4 hours practical work devoted to the illustration of superalloys creep at elevated temperatures.

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