DEFORMATION AND FRACTURE – LAB COURSE

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The creep test

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1. Introduction

1.1 Overview

- Creep is defined as a *time-dependent plastic deformation that occurs at constant stress and temperature*. It is due to the inelastic response of loaded materials at high temperatures.
- Elasticity and plasticity are mechanical responses to loading which are independent of time. As soon as the load is applied, the corresponding level of strain sets in. In contrast, during creep the mechanical response is time dependent. This is somewhat analogous to viscoelastic behavior except that during creep an often significant portion of the strain is permanent and remains after unloading.
- Temperature has important effects on deformation phenomena. Microstructural defect rearrangements are often accelerated at high temperatures. Since these processes tend to soften the material, they counteract the strain hardening produced by plastic deformation.
- The point, then, is that the temperature at which materials start to creep depends on their melting point. As a general rule, it is found that creep starts when
  $T > 0.3$ to 0.4 T_m for metals and alloys,
  $T > 0.4$ to 0.5 T_m for ceramics.
- The first requirement for a creep resistant material is therefore that it feature a high melting (or softening) temperature. If the material can then be used at less than 0.3 of T_m, creep will not be a problem. If it has to be used above this temperature, various alloying procedures and heat treatments can be used to increase the creep resistance.

1.2 Examples of creep deformation

- Tungsten lamps run at about 2000 °C—this, for tungsten, is a high temperature. If you examine a lamp filament that has failed, you will see that it has sagged under its own weight until the turns of the coil have touched—that is, it has deformed by creep.
- Lead, for instance, has a melting point of 600 K; room temperature, 300 K, is exactly half its absolute melting point. Room temperature for lead is a high temperature, and therefore lead may experience creep. The same holds for many solder alloys.
- 'Temperate' glaciers (those close to 0 °C) are at a temperature at which ice creeps rapidly—that is why glaciers move. Even the thickness of the Antarctic ice cap, which controls the levels of the earth's oceans, is determined by the creep-spreading of the ice at about -30 °C.
- High-temperature creep is of major concern in such applications as jet engines or nuclear reactors. Bolts in jet engines or blades fail mainly due to creep whether nuclear fuel rods can break or deform beyond repair because of this effect.

1.3 Types of creep failure in applications

- Displacement–limited applications, in which precise dimensions or small clearances must be maintained (as in the discs and blades of turbines).
- Rupture–limited applications, in which dimensional tolerance is relatively unimportant, but fracture must be avoided (as in pressure–piping).
- Stress-relaxation–limited applications, in which an initial tension relaxes with time (as in the pretensioning of cables or bolts).
- Buckling–limited applications, in which slender columns or panels carry compressive loads (as in the upper wing skin of an aircraft, or an externally pressurized tube.

1.4 Aim of this lab

The aim of this lab is to study the mechanical behaviour of a (metallic) material at "elevated" temperature. In industrial practice, it is important to know the material characteristics that enter the constitutive laws that describe the mechanical behaviour of materials at elevated temperature. This is important not only for the dimensioning of structures, but also for predicting their lifespan. Most material characteristics that affect these laws can be determined by a creep test. The purpose of this lab is to present the creep test, to learn how to read a creep curve and to determine certain material parameters that govern creep.

2. Theory

2.1 Creep rate

Even though plastic deformation in metallic materials is mostly driven by dislocation multiplication and glide, this changes gradually with an increase in temperature. These changes occur mostly above the softening temperature of a material, which usually is between 0.3 and 0.5 of the melting temperature (in degrees Kelvin). Above this temperature, thermal activation becomes important enough to significantly assist dislocations for passing obstacles.

Thus work hardening, which hinders and eventually impedes low temperature plastic deformation if an applied load remains below the fracture load, does not operate the same way at high temperatures. On the one hand, at elevated temperature recovery and recrystallization anneal rapidly an important proportion of work hardening. On the other hand, when a sample is maintained under load, thermal activation helps dislocations to overcome the Peierls potential and thus increases their mobility.

Characteristic for a typical creep curve is a relatively high creep rate immediately after the loading of the sample (primary creep stage, stage I, Fig. 1). At this stage, dislocations can move freely until major obstacles stop them. Then, the creep rate slows down substantially and reaches a steady state, i.e. becomes independent of time and of prior deformation (secondary creep, stage II). This steady state is due to equilibrium between work hardening and structural rearrangement (recovery and recrystallization), as stated above. For numerous materials, especially for pure metals and ionic crystals, this stage of constant (steady state) creep rate is maintained for a relatively long time. However, a majority of industrial alloys never attain steady state creep (absence of stage II). Those materials still exhibit a minimum of creep rate before a slow increase set in, which results from the accumulation of microstructural damage. During tertiary creep (stage III), the creep rate increases until fracture.

Thermal activation does not completely anneal all work hardening. Especially when the creep rate increases because of a large imposed stress, more dislocations are produced than annihilated. The dislocation density then increases. As already stated, however, this does not translate into a homogenous hardening that will eventually stop any further deformation. In general, microstructural observations show that dislocations, initially arranged in a homogenous network, start to organize themselves. They first form a cellular structure; then, due to the effect of recovery, these cells slowly transform into subgrains.

The interior of these cells and sub-grains contains only few dislocations, such that these regions are comparably soft. On the other hand, the cell walls turn into subgrain boundaries and become more and more impenetrable to dislocations. Dislocation nucleated inside these cells can easily glide to the walls, where they become trapped. The walls, however, resist plastic deformation up to much higher stresses. Rearrangements of dislocations in the walls capable of transmitting the deformation from one subgrain to another do not occur instantaneously. It is particularly dislocation climb, governed by thermal activation, which now limits the rate of

deformation. In this state, the creep rate is at its minimum or stationary value. Indeed, observations show that the subgrains change shape for a long time without their size changing (equilibrium between work hardening and recovery). It can be easily understood that an externally applied load will concentrate on the subgrain boundaries. The ongoing excess of stress acting on the subgrain boundaries causes microstructural damage, which weakens the metal. The creep rate finally increases and the sample fractures when damage becomes critical.
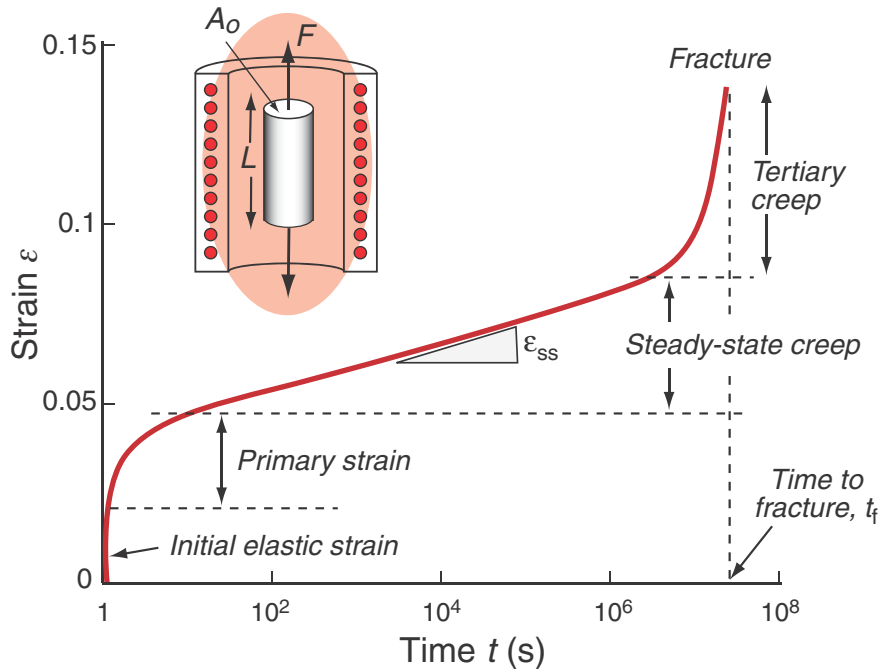

Figure 1. Different stages of creep (Ashby et al.).

The description given above is only one mechanism among others. Indeed, at temperatures that approach the melting temperature, stress assisted diffusion of atoms and vacancies becomes important. Also, grain boundary sliding has to be considered. Apart from the dislocation network and the subgrains, other microstructural parameters, such as precipitates and dispersed particles, as well as grain size strongly affect the creep rate.

2.2 Phenomenological laws and coefficients

The analytical description of a creep curve as a function of time or strain, which is based solely on elementary mechanisms, is not easy at all. At least the microstructural details (metal, precipitates, grain size, etc.), as well as the contribution of different mechanisms (dislocations, diffusion, grain boundaries) have to be accounted for. In practice, in most cases, measured curves are used. To estimate the effect of stress σ and temperature T, one focuses generally on stage II (constant creep rate). For those materials that do not exhibit a distinct stage II, the minimum

creep rate is used. Consideration of the constant creep rate stage is justified by the fact that it often represents a substantial part of the lifespan of a part. Stage I only lasts a short time, and stage III is not of engineering interest, since the useful lifespan of a structural part has expired by then and the part has to be replaced for safety reasons. In the following, we focus on stage II.

The steady state creep rate $\dot{\varepsilon}_{ss}$ is a function of both stress (a) and temperature (b), as shown in Fig. 2.
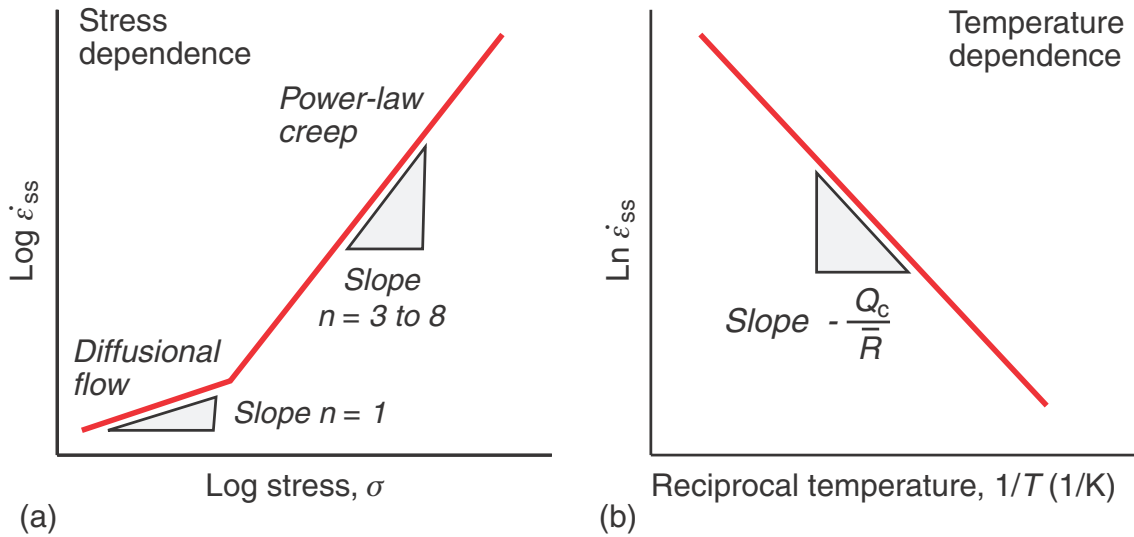

Figure 2. Stress and temperature dependence of the steady state creep rate (Ashby et al.).

The stress dependence can be described as

$$\dot{\varepsilon}_{ss} = B \cdot \sigma^n \text{,}$$

(1)

the temperature dependence as

$$\dot{\varepsilon}_{ss} = C \cdot \exp\left(\frac{-Q_c}{RT}\right).$$

(2)

Combining gives for the minimum creep rate (i.e., in steady state creep, stage II),

$$\dot{\varepsilon}_{ss} = A \cdot \sigma^n \cdot \exp\left(\frac{-Q_c}{RT}\right)$$

(3)

where A: constant, σ: stress [MPa], n: exponent, Q_c: activation energy [J/mol], $R = 8.314$ J/mol·K (ideal gas constant).

When the applied stress is not too high, this law usually gives a satisfactory description of the creep rate as a function of temperature and applied stress. The constant A, the exponent n, and the activation energy for creep Q_c are material characteristics and can be determined from a series of creep tests. Several tests have to be carried out since temperature has to be varied at constant applied stress in order to determine the activation energy, and stress has to be varied at constant temperature to determine the exponent n. This is consequently always a long term project. The phenomenological law is not precise enough to calculate (by extrapolation) the creep rate at low temperatures from measurements taken at high stress near the melting temperature, where things happen very fast.

The exponent n can be easily determined through a double-logarithmic representation of the creep rate (at $T = $ constant) and the stress. When the observed results satisfy the law mentioned above, a straight line of slope n should be obtained. Similarly, the logarithm of the creep rate (at constant stress) can be plotted as a function of $1/T$. The slope of the straight line should equal Q_c/R. For pure metals only a few control measures need to be taken, since it is well established that the activation energy for creep corresponds to the activation energy for auto-diffusion. This fact is also confirmed by theory, since the stationary creep rate is limited by rearrangements that require the climb of dislocations in the subgrain boundaries.

In pure metals, the exponent n varies between 4 and 5 (Norton power law). For single phased alloys (solid solutions), n equals rather 3. In these two metal groups, results obtained at very high temperatures show an exponential dependence of stress such that the creep curve deviates from Norton's law. A very strong dependence can also be observed in precipitate-hardened alloys. It is not unusual that n has to be chosen above 10 to describe the experimental results, even at low stress. In order to "save" Norton's law with reasonable exponents (3–5), one may argue that solely the difference between applied stress and the back-stress induced by the precipitates is available for driving creep deformation. The applied stress in Norton's law should then be replaced by the "effective" stress, which corresponds to this difference.

3. Creep testing

3.1 Creep machines and creep tests

During a creep test, plastic deformation is measured as a function of time, at constant temperature and at a constant applied stress. The different deformation modes that can be imposed are tension, compression, bending and torsion. For compressive testing, samples of very simple shape can be used. It is notably convenient for materials that are brittle and difficult to machine. In torsion testing large strains can be reached before fracture. This type of test is usually used to assess the suitability of a material for forming processes. To calculate the resistance of a structure exposed to high temperatures, typically tensile tests are required. Machines for tensile creep testing

resemble conventional tensile machines, Fig. 1. Since they serve essentially as "sample-holders", their design is quite basic. Instead of a moving crosshead, a weight is applied at the beginning of the test as load, and instead of hydraulic grips, tapered or threaded samples are used. At the most elevated temperatures, diffusion welding between the sample and the fixation may occur. To avoid this, lubricants resisting to high temperatures are used, for example graphite or $MoSi_2$. Obviously the interior of the furnace has to be resistant to high temperature and oxidation. Highly alloyed steels and superalloys are the preferred materials. To determine the precise elongation, an extensometer is required, just like for the classical tensile test.
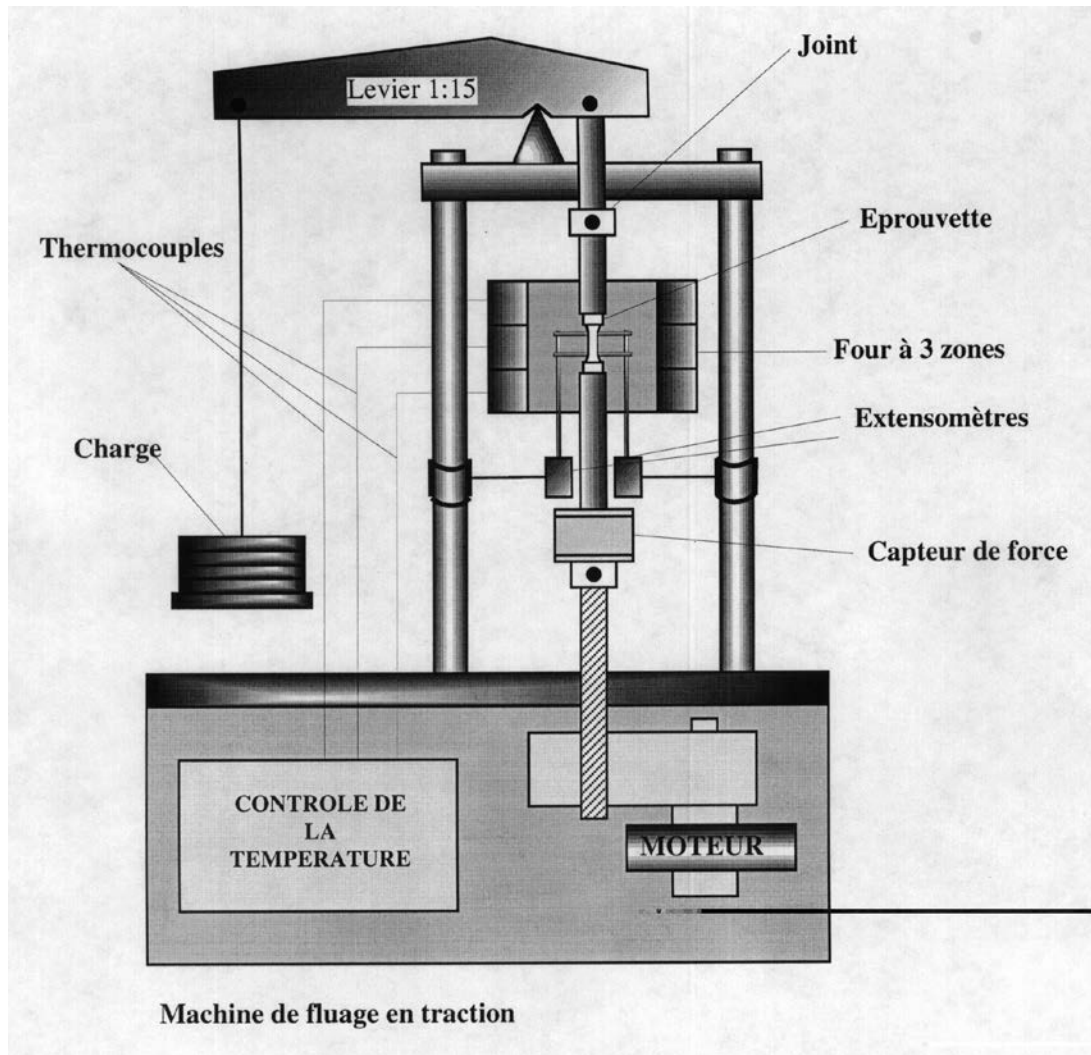

Figure 3. Creep frame.

Before finishing this section on creep machines, it should be mentioned that simple machines as described above operate at constant load rather than constant stress. This agrees perfectly well with the needs of industry, where one encounters rather situations where a load is imposed. In fundamental research, however, creep curves obtained at constant (true) stress are preferable.

They facilitate theoretical interpretation, which is the base of any improvement of creep resistance. Indeed, the driving force for creep is always a "local effort", stress, density of elastic energy or Peach-Köhler force. For small deformations (1%), the difference between constant load and constant (true) stress affects the creep rate barely and stays negligible. However, when the deformation reaches 20%, the effective stress σ increases by $(1+e)_\sigma = 1.2$ (where e designates the engineering strain) and according to Norton's law the true creep rate increases by $\dot{\varepsilon} \propto \sigma^n \propto (1+e)^n$. This last factor equals 2.07 for $n = 4$. These differences cannot be ignored any more, and thus a mechanism that compensates the increase of stress with the reduction of the cross section of the sample is needed.

3.2 Specimens and material

In order to be able to measure creep curves at different applied stresses during the very short duration of the lab course, we have to work at ambient temperature and use a metal with a low melting temperature. Lead is a metal that fulfils perfectly this condition. It has a melting temperature of 327 °C and thus ambient temperature corresponds already to 0.5 of the melting temperature. Recovery and recrystallization indeed already start at temperatures below 0 °C and we can thus observe classical creep behaviour at 20 °C.

The shape of the creep test specimens is inspired by standard tensile test samples. The diameter is usually between 5 and 10 mm, and the ratio between the sample length and its diameter should be larger than 5. Since lead casting is much easier than machining, the specimens are cast into steel moulds. The specimen geometry is indicated in Fig. 3.
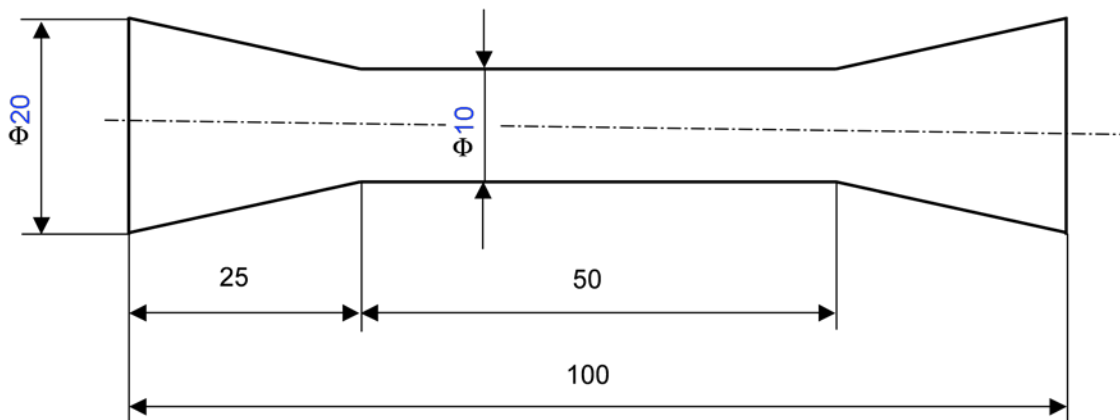

Figure 4. Specimen geometry. All dimensions are in mm.

4. Experimental procedures for this lab

4.1 Casting of the specimens

Lead is melted in a crucible and heated to 340 °C in a furnace. To avoid oxidation, lead should not be heated above 350 °C. A protective, or even reductive, atmosphere is recommended. A perfect atmosphere for preventing oxidation is forming gas (a mixture of nitrogen with roughly 5% of hydrogen). For casting, a stainless steel mould that is placed on top of a copper plate is used. The solidification front then advances from the bottom to the top and cannot trap any liquid lead. Otherwise shrinkage pores might result, because lead reduces its volume by 3.6% upon solidification.

4.2 Creep testing

Several creep curves are to be established by varying σ and T in order to calculate the parameters A, n and Q_c of Norton's law.

5. Bibliography

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