

# Creep and Superplasticity at a glance

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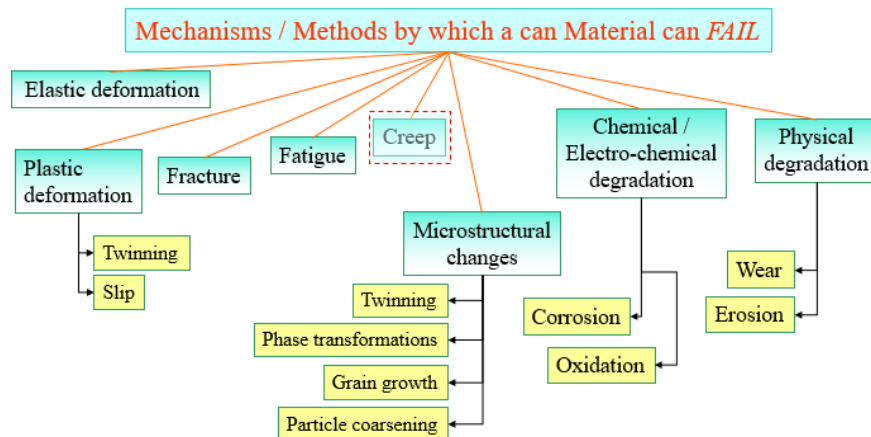
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**Abstract:** One of the most difficult tasks a material scientist must complete is designing materials for high temperature applications. The desired microstructure tends to deteriorate as a result of several thermodynamic and kinetic variables. This is due to the fact that diffusion, among other underlying processes, has a temperature-dependent exponential kinetics. Therefore, in this chapter we will mainly discuss the creep and superplasticity phenomena to design the material for different potential applications. Further, factors affecting the creep, effect of stress and temperature on the creep curve, creep mechanism of crystalline material, creep resistant material, and creep in nanomaterials are discussed in detail. Creep is defined as permanent distortion (plastic deformation) of a material over time under a constant load or stress. In certain ways, the phenomena of creep and superplasticity are related: in the case of creep, damage accumulation can result in sample failure, but in the case of superplasticity, extended plastic deformation can be accomplished (damage accumulation leading to failure is postponed). Moreover, we have illustrated the superplastic materials, which shows the extremely large tensile elongation before failure. Furthermore, we have outlined the superplasticity mechanism in polycrystalline material, and in nanomaterials.

**Keywords:** Failure mechanism, Creep mechanism, Superplasticity.

## 1. Introduction

Development of materials for high temperature applications is one of the most challenging tasks for a material scientist. Several thermodynamic and kinetic factors might cause the ideal microstructure to degrade. This is because, among other underlying processes, diffusion has an exponential kinetics that is temperature dependent. Therefore, even a slight rise in temperature can have "catastrophic" effects. High temperatures lead to a reduction in strength and an accumulation of material damage (such as void formation).



**Fig 1. Different failure mechanisms in materials.**

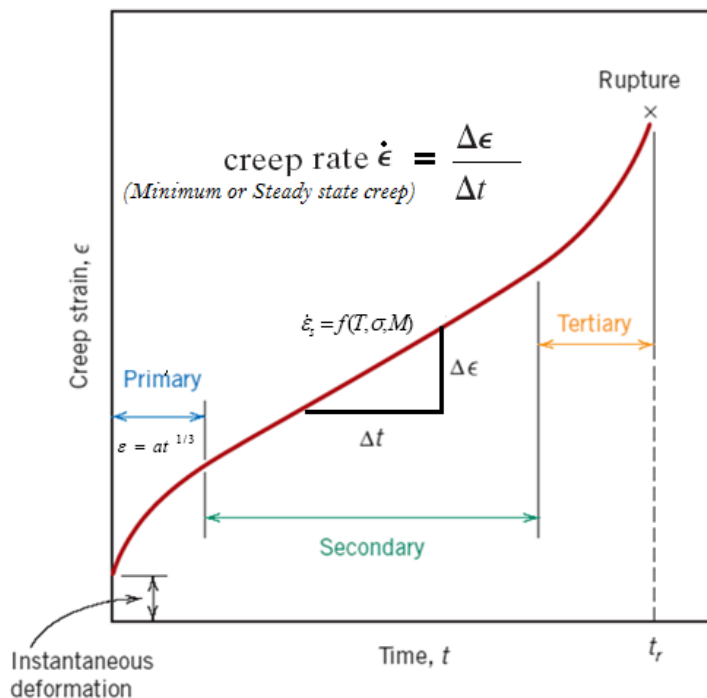
Creep is described as the gradual plastic deformation (permanent distortion) of a material under a steady load or stress [1,7]. Superplasticity is the capacity of a polycrystalline material to display extremely large tensile elongation before failure in a generally isotropic way [1,2]. Fig.1. shows the failure of the material with different mechanisms and methods. In this chapter we will mainly discuss the creep phenomena. Creep is the term for time-dependent plastic deformation of materials under a constant load or stress. Due to fact that this scenario most closely resembles actual service loading circumstances, constant load testing is important for engineering objectives. Continuous stress testing, on the other hand, is helpful in gathering fundamental data that may be used in process of discovering deformation mechanisms. After loading, the strain grows over time until failure happens.

## 2. Creep and associated mechanism

In this section we will discuss factors affecting the creep, effect of stress and temperature on the creep curve, creep mechanism of crystalline material, creep resistant material, and creep in nanomaterials

### 2.1 Creep Curve

The creep curve (strain vs. time) often exhibits three stages following the first instantaneous strain: a primary stage, a secondary stage, and a tertiary stage. The strain rate in initial stage goes down over time. The secondary stage, often referred to as the steady-state stage, is when strain rate either reaches a minimal value (corresponding to constant load) or stays constant throughout time. The strain rate quickly rises over time in the tertiary stage. (Fig 2). The steady-state strain rate as a function of applied stress, temperature, and grain size is essential for determining the mechanism of deformation that governs the creep process.



**Fig 2. Typical creep curve showing 3 stages of creep.**

- I-Primary creep or Transient creep
  - Decreasing creep rate due to strain hardening until reaches a constant value
  - Observed in all temp: (cold creep)

- II-Secondary creep
  - Constant creep rate, due to the balancing between strain hardening and recovery effects
  - Creep takes place by viscous flow in materials.
  - Hot creep (happens at high temp)
- III-Tertiary creep
  - Due to necking, increasing creep rate
  - Final fracture failure

Creep is found to occur at higher temperatures than at lower temp. Therefore, study of creep is very important for those materials which are used at high temp like components of gas turbines (800 to 950K), Burner and after burner (1300 to 1400K), Nuclear reactor (650 to 950K), furnaces, rockets, missiles etc. In polymers and glass, Creep is important when  $T > T_g$  (Glass Transition Temperature), at this temperature, those become rubbery and viscoelastic or viscoplastic, Lead creeps at ambient temp, Iron creeps at 600°C, Nickel based super alloys at 1227°C, Ceramics at 2000°C.

### 2.2 Factors affecting the creep

- Load
  - Creep rate increases as load increases, Time to rupture decreases
- Temperature
  - Temp increases- mobility of atoms and vacancies increases which speeds up the diffusion, Creep rate increases as T increases
  - Creep rupture life decreases
- Composition
  - Hence creep is more in pure metals
  - In alloys, different phases present stops dislocation glide
- Grain size
  - Smaller grain, stronger material.
  - But above Equicohesive temp. this effect will be reversed one.
  - Equicohesive temp. (Kelvin)  $T_e > T_m/2$
- Heat treatment
  - This changes structure, obviously materials property changes, creep resistance also changes.

### 2.3 Effect of stress and temperature on creep curve (constant load)

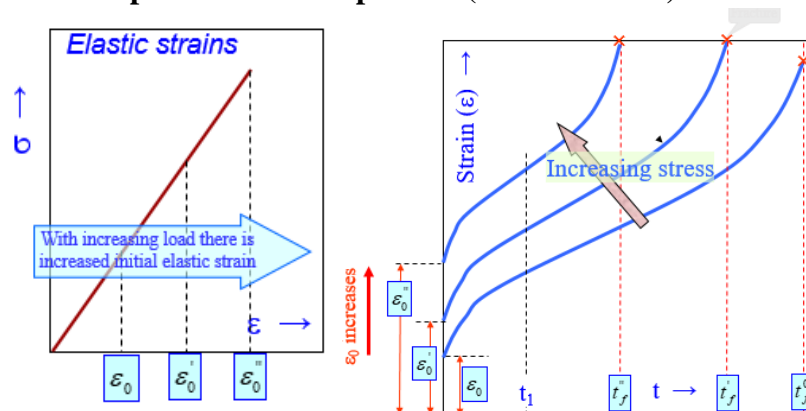
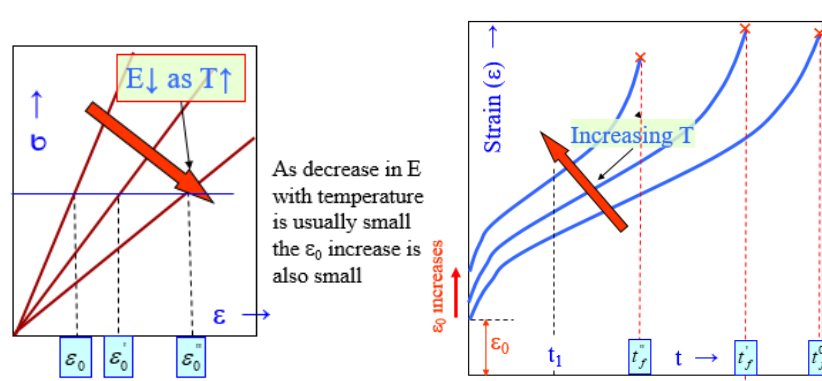


Fig 3. Effect of stress on creep curve.

As the load for the experiment is increased (Fig.3), three things happen: (i) the elastic instantaneous strain increases, (ii) the elastic strain is greater for a given time (say,  $t_1$ ), and (iii) the time to failure ( $t_f$ ) reduces (i.e., as expected, specimens fail earlier).

#### Effect of temperature

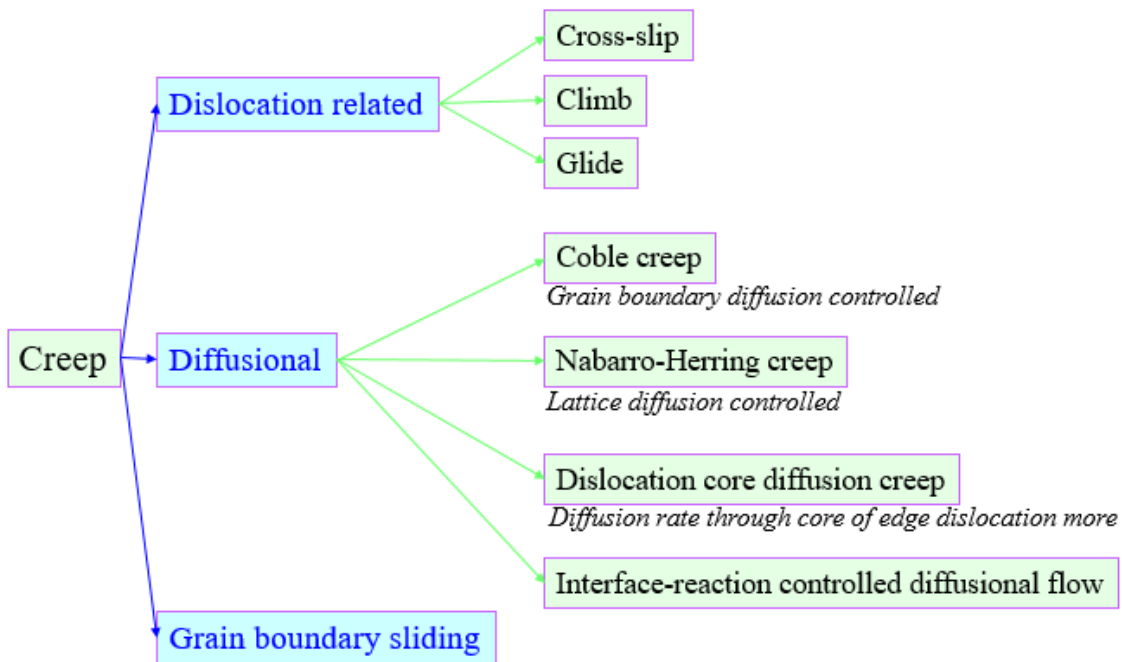
- ❑ When temperature at which experiment is run is raised (Fig. 4), following effects occur: (i) instantaneous strain (elastic = 0) increases (slightly); (ii) strain is greater for a given time (say,  $t_1$ ); and (iii) time to failure ( $t_f$ ) shortens.
- ❑ The instantaneous strain  $\epsilon_0$  increases with increasing  $T$  because of slight decrease in Young's modulus ( $E$ ) of material.



**Fig 4. Effect of temperature on creep curve.**

## 2.4 Creep mechanism of crystalline material

- The two key factors that influence both the mechanism's operation and the creep rate are stress and temperature. Three different types of mechanisms: dislocation-related, diffusional, and grain boundary sliding—are at work during creep (Fig.5). Further, we display these along with their subclasses.
- Due to shear stress, two grains can slide past one another at high temperatures because grain boundary is weaker than the grain core. The term "equicohesive temperature" refers to temperature at which grain is as strong as grain border.
- It's also possible that a combination of these methods causes the creep strain.
- Other modes of plastic deformation or microstructural changes may take place simultaneously with creep, depending on the load and temperature. These include dynamic recrystallization and plastic deformation by slip.
- With homologous temperature ( $T/T_m$ ) & normalized shear stress ( $\tau/G$ ) as axes, deformation mechanism maps can be created. Other combinations of variables, such as  $T/T_m$  vs. shear strain rate or normalized shear stress vs. shear strain rate, may also be used for these plots. These maps frequently superimpose descriptors that are based on both phenomenology and mechanism.



**Fig 5. Creep mechanism in crystalline material [7,8,9].**

➤ **Dislocation related mechanisms**

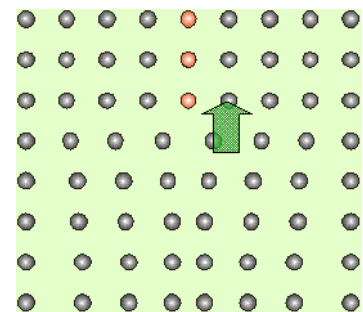
- Regarding the activity of dislocations, two roles may be distinguished: (i) it is the main source of strain, and (ii) it plays a secondary role to accommodate local strain (while main source of strain is another mechanism, such as grain boundary sliding).

Cross-slip

- Low temperatures are when this type of creep is seen. Dislocations in this screw cross-slip due to thermal activation and produce plastic strain as a function of time.

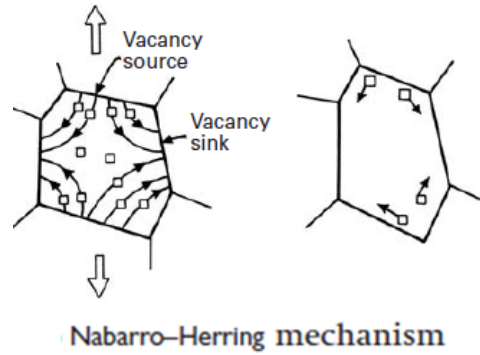
Dislocation climb

- Edge dislocations that are stacked up against a barrier have the potential to climb to another slip plane & result in plastic deformation. This results in strain as a function of time as a reaction to stress. It should be emphasized that these dislocations (which are being pinned) are sessile at low temperatures and only turn glissile at high temperatures.
- Rate controlling step is diffusion of vacancies.



➤ **Diffusional creep**

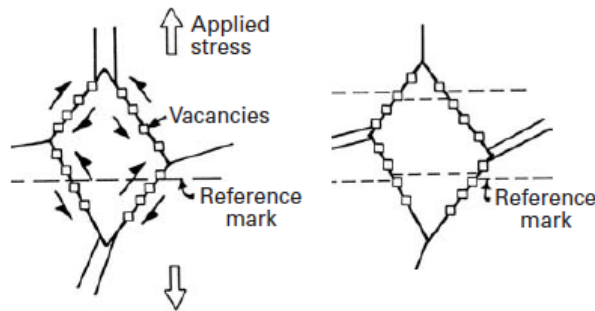
It happens at high temperatures and under relatively light forces. The creep rate is regulated by the diffusion of vacancies. As a result, vacancies move from the compression zone to the tensile region while atoms move in the other way. i.e., producing creep strain along the tensile axis. From the top and bottom to the lateral areas of the grain, voids in the grain move.



Nabarro-Herring mechanism

**Coble creep**

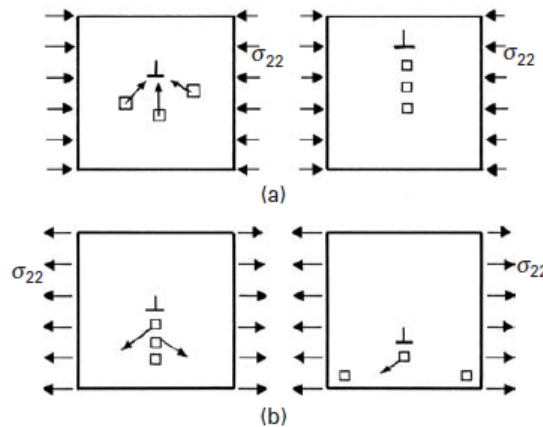
Diffusion in the grain boundaries instead of bulk resulting in the sliding of grain boundaries. For high creep resistance, go for larger grain size (super alloys)



Coble mechanism

**Harper Dorn creep**

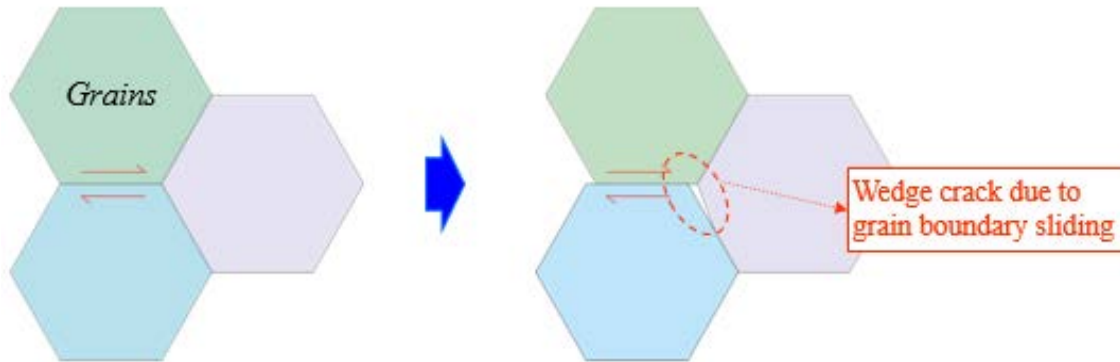
- Compressive stress causes voids to be drawn near the dislocation line.
- The dislocation line is successfully translated upward once a row of voids has connected.
- Reverse happens in tension



➤ **Grain boundary sliding**

- The movement of dislocations is hampered at low temperatures because grain boundaries are "stronger" than crystal core.
- Being a higher energy region, grain boundaries *pre-melt* before crystal interior.

- Grain boundaries slip past one another above the equicohesive temperature as a result of shear stress at the "local scale" causing plastic deformation.
- At triple lines (the intersection of three grains), relative motion of grain boundaries can result in wedge cracks (Fig.6). In the event that these wedge cracks are not repaired by diffusion (or slip), microstructural damage will build up & the specimen will fail.



**Fig 6. Grain boundary sliding**

### 2.5 Phenomenological descriptions of creep

One of the important descriptions of creep is using the power-law formula. The shear strain rate is a power function of the shear stress. Clearly this formula is not based on a mechanism operative, but a fit of data.

$$\dot{\gamma} = \left( \frac{\tau}{G} \right)^n$$

**Where,**

$\dot{\gamma}$  → Shear Strain Rate

G → Shear Modulus

$\tau$  → Shear Stress

n → An exponent having a value between ~ 3-10

Power-law behavior can arise from:

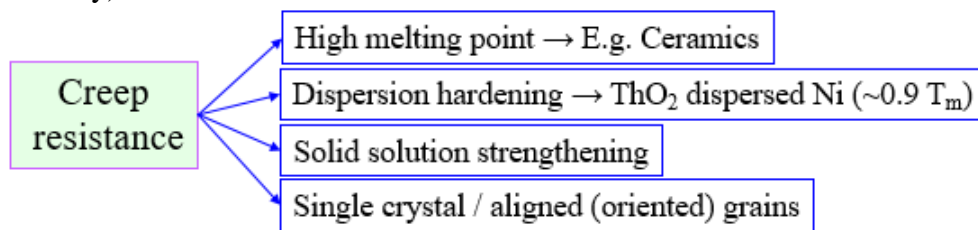
- Only glide at low temperatures ( $\sim 0.3T_M$ ). Here exponent  $n \sim 3$ .
- Glide + climb (referred to as climb-controlled creep) occurs at higher temperatures. Above  $\sim 0.6T_M$  climb is lattice-diffusion controlled. At low temperatures than this pipe diffusion may play an important role in creep.
- At high stresses ( $> 10^{-3}G$ ) power law breaks down. At high stresses mechanism changes from climb-controlled (creep) to glide-controlled (slip).

### 2.6 Creep resistant material

- The demand for materials that can operate at high temperatures (and in some applications for extended periods of time) is rising. For a heat engine, higher operating temperatures result in

greater efficiency. As a result, it is necessary to create materials that can endure extreme temperatures.

- It should be emphasized that materials should have good oxidation resistance and other qualities for use in high temperature applications. Cost, ease of manufacture, density, and other factors are crucial in deciding ultimate material selection.
- Some material design techniques, such as work hardening, precipitation hardening with coarsening precipitates, particle size reduction, etc., that are effective at low temperatures are ineffective at high temperatures.
- A few effective methods include: (i) aligning grain boundaries with the main loading axis, (ii) producing single crystal components, such as turbine blades; (iii) using dispersoids for strengthening, (iv) using precipitates with low interfacial energy to strengthen (which will not coarsen easily).



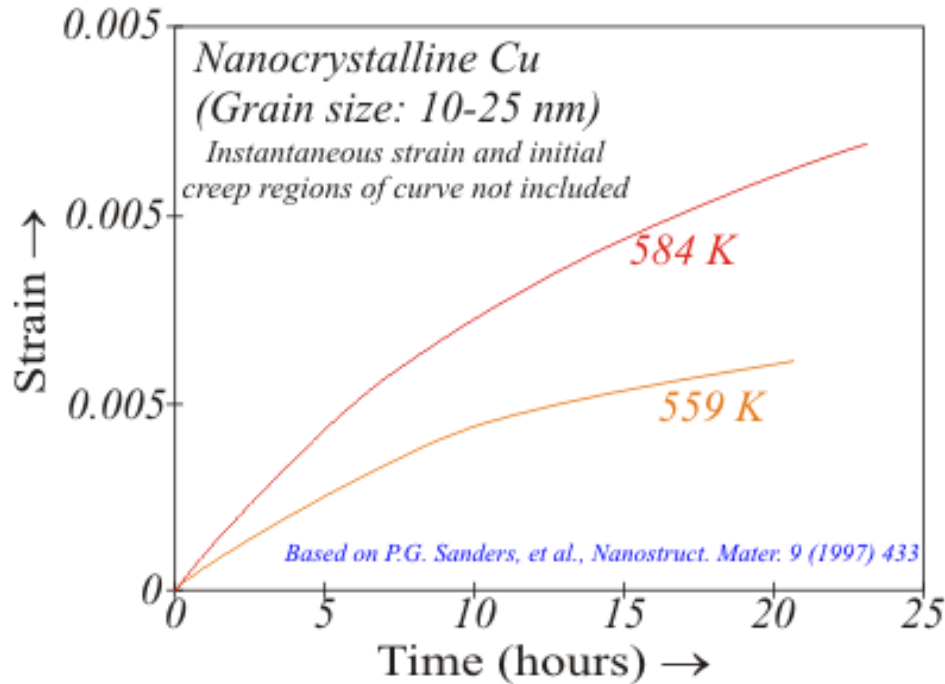
- Materials frequently used include Fe, Ni (including super alloys), and Co based alloys.
- The "usual precipitates" approach to precipitation hardening is not a good one since the precipitates coarsen (smaller particles dissolve & larger particles increase, causing inter particle separation increases and a reduction in strength).
- Ni<sub>3</sub>(Ti,Al) precipitates form a low energy contact with matrix in Ni-base superalloys. This lessens the power that drives coarsening. (Note: Other activities like rafting may cause the characteristics of such materials to degrade).
- Due to the possibility of recrystallization, which results in strain-free crystals, cold work cannot be employed to increase creep resistance.
- Contrary to what is typically done to increase the low temperature strength, fine grain size is not desired for creep resistance because grain boundary sliding can produce creep elongation/cavitation. Consequently, the following two tactics are applicable.
  - ▶ Use single crystals (single crystal Ti turbine blades in gas turbine engine have been used—though they are very costly).
  - ▶ Aligned/oriented polycrystals → as all grain boundaries are aligned along primary tensile axis, they experience no shear stress and creep is negated.

## 2.7 Creep in Nanomaterials

Numerous unique characteristics of nanocrystalline materials have been demonstrated. Numerous studies on mechanical behavior have been published in literature, the majority of which focus on the relationship between grain size and room temperature microhardness. Nieman, Weertman, and Siegel [12] investigated the tensile strength and constant load creep of nanocrystalline Pd samples produced by inert-gas condensation at ambient temperature. Their findings suggested that grain boundary diffusion did not play a role in creep at ambient temperature [10,11].



- Because of their tiny grain size, nanostructured materials (grain size in nanoscale zone) are expected to display creep at lower temperatures, greater creep rates for a given temperature, and a preponderance of processes such as grain boundary diffusion & grain boundary sliding. Now we can look at what the experiments truly show.
- When compared to materials with micron-sized grains, the creep rate in nanocrystalline Pd (~40 nm) and Cu (~20 nm) did not appear to be higher (and for Pd, a lower creep rate was reported in particular temperature regimes). The idea that nanocrystalline materials will experience a higher creep rate is directly at odds with this.

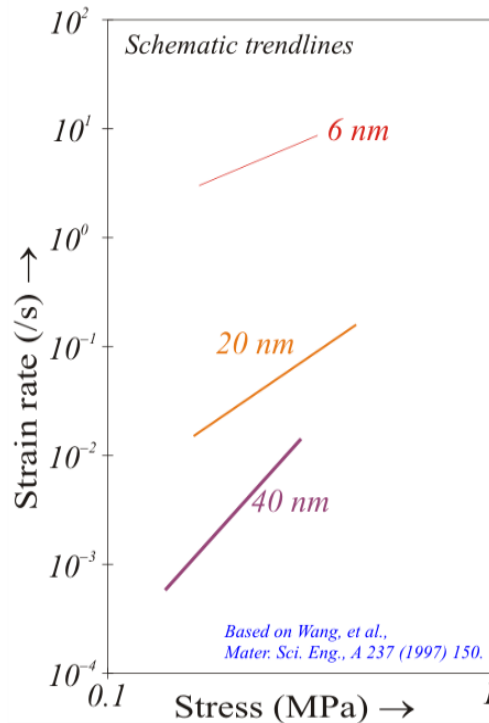


- Studies on Cu (10-25 nm GS), Pd (35-55 nm GS) (TEM showed porosity in sample)
  - Creep in low T regime ( $0.24-0.33 T_m$ ) → low creep rate, low grain growth
  - Creep in medium T regime ( $0.33-0.48 T_m$ ) → creep rate decreasing even after long testing time, grain growth (25 nm → 100s of nm)

In some instances, the creep rate in the nanoscale regime of grain sizes rose as the grain size decreased (for example, in Ni-P nanocrystalline material, creep rate of material with ~30 nm particle size was higher than that of material with 250 nm grain size).

In cases where high creep rate expected for nano crystalline materials (e.g. Pd, Cu) was not observed, reason attributed are:

- presence of low angle grain boundaries and twin boundaries (which are not prone to sliding and have low diffusivity for vacancies),
- reduced dislocation activity in nanocrystalline samples



- Creep of nc-Ni at RT (Grain Size (GS): 6, 20, 40 nm):
  - Smaller grain size (6nm) showed faster creep rate.
  - Behaviour consistent with Grain boundary sliding controlled by grain boundary diffusion mechanism.
  - At high stresses and larger Grain Size (20, 40 nm), dislocation creep was observed.

### 3. Principal of Superplasticity

Superplasticity is capacity of a polycrystalline material to display extremely large tensile elongations before to failure in a generally isotropic way ( $T > 0.5 T_m$ ) [1,2]. As early as 1912, observations of this phenomena were made. Since that time, metals have been the subject of in-depth research on superplasticity [3,4]. Superplastic materials are thought to have been present in both arsenic bronzes used in Turkey during Bronze Age (2500 B.C.) and Damascus steels used from 300 B.C. to end of nineteenth century. Perhaps Pearson's 1934 report on a Bi-Sn alloy that experienced approximately 2000% elongation is one of most spectacular observations of superplasticity. For the first time, he also asserted at that time that the primary deformation mechanism causing superplastic deformation was grain-boundary sliding. Recent observations of this phenomena in a variety of materials, including some that are challenging to form by traditional forming (such as nanocrystalline materials, ceramics, metal matrix composites, & intermetallics), have increased interest in superplasticity. Fine structure superplastic materials generally exhibit a high strain-rate sensitivity exponent ( $m$ ) during tensile deformation. Typically,  $m$  is larger than 0.33.

The phenomenon of extensive plastic deformation without necking is termed as structural superplasticity. Superplastic deformation in tension can be  $>300\%$  (up to even 2000%).

- Typically, superplastic deformation occurs when:
  - (i)  $T > 0.5T_m$
  - (ii) grain size is  $< 10 \mu\text{m}$
  - (iii) grains are equiaxed (which usually remain so after deformation)
  - (iv) grain boundaries are glassy (with a large fraction of high angle grain boundaries).
- The compositions of many superplastic alloys are almost eutectic or eutectoid in nature.
- Diffusion can be controlled at the grain boundary or the lattice in superplastic flow.
- A plot of stress versus strain rate is often sigmoidal and shows 3 regions (Fig.8):

- Low stress, low strain rate regime in Region-I ( $\dot{\epsilon} < 10^{-5} /\text{s}$ ), where,  $m \in (0.2, 0.33)$ . Sensitive to sample's purity. Lower ductility & dispersion across the grain boundaries.
- Intermediate stress and strain rate regime in region II

$[\dot{\epsilon} \in (10^{-5}, 10^{-2})]$ , where  $m \in (0.4, 0.67)$ . Extended region with a range of strain rates exceeding several orders of magnitude. maximal ductility zone. Grain size and purity are not factors that affect strain rate.

commonly known as superplastic zone.

Mechanism → predominantly grain boundary sliding accommodated by dislocation activity (Activation energy (Q) corresponding to grain boundary diffusion ( $Q_{gb}$ )).

- Region-III- high stress and strain rate regime ( $\dot{\epsilon} > 10^{-2} /\text{s}$ ) →  $m > 0.33$   
Creep rates sensitive to grain size.

Mechanism → intragranular dislocation process (interacting with grain boundaries).

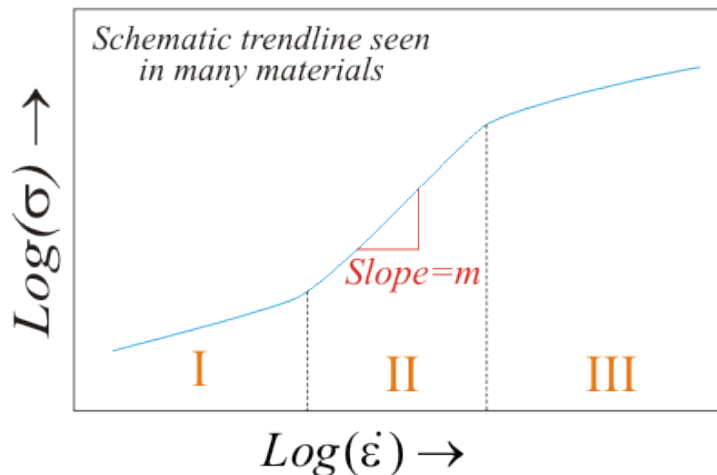


Fig 8. Sigmoidal curve

### 3.1. Superplasticity in nanomaterials

Superplasticity, or a material's capacity to withstand significant plastic deformation, has been proven in a number of metallic, intermetallic, & ceramic systems. A stable fine-grained microstructure and a temperature greater than  $0.5 T_m$  (where  $T_m$  is melting point of matrix) are prerequisites for superplasticity. Low-temperature superplasticity in nanocrystalline nickel, a nanocrystalline aluminium alloy (1420-Al), & nanocrystalline nickel aluminide ( $\text{Ni}_3\text{Al}$ ) are presented by McFadden et al. [13]. The nanocrystalline nickel was discovered to be superplastic at a temperature  $470^\circ\text{C}$  lower than previously attained: this

equates to  $0.36 T_m$ , lowest normalized superplastic temperature known for any crystalline material. The nanocrystalline  $Ni_3Al$  was shown to be superplastic at a temperature  $450^\circ C$  below superplastic temperature in microcrystalline regime.

- Most often, superplasticity has not lived up to the early 'expectations'.
- Superplasticity is frequently only seen in nanocrystalline samples, despite the fact that it is also seen in their microcrystalline counterparts.
- At  $0.36T_m$  (more than  $450^\circ C$  lower than the temperature for bulk material), superplasticity was seen in nanocrystalline Ni (20 nm particle size).
- About  $450^\circ C$  less than its microcrystalline counterparts, nanocrystalline  $Ni_3Al$  (grain size 50 nm) also underwent superplastic deformation. At  $650^\circ C$  and a strain rate of  $10^{-3}$  /s,  $Ni_3Al$  exhibited a ductility of 350%.
- Superplasticity was demonstrated by the 1420-Al alloy at a high strain rate of  $10^{-1}$  /s. These situations result in significant work hardening & higher flow stresses for superplastic deformation compared to materials with micron-sized grains.

Increased diffusion, grain boundary sliding, & dislocation activity led to superplasticity at low temperatures (or, alternatively, superplasticity at high strain rates ( $\dot{\epsilon} > 10^{-2}$  /s) at a given temperature in superplastic regime).

A significant problem in superplasticity tests is grain growth. From a starting grain size on order of 20 nm, it was seen in the instance of nc-Ni that grain size could grow to 1 micron levels. The development of the grains may be less in other materials. Intermetallic compounds and two-phase mixtures (with the second phase being favored as a precipitate) are predicted to have less grain growth. The second phase in two-phase mixtures pins the grain boundaries, whereas intermetallic (such  $Ni_3Al$ ) require order to be maintained during grain growth in order to prevent the process from becoming uncontrolled.

Long-range stress fields related to non-equilibrium boundaries of grains are expected to impede grain border sliding, so non-equilibrium grain boundaries give lower elongation than equilibrium grain boundaries in circumstances where grain boundary sliding is the dominant mechanism for the superplasticity (for example, in some Mg alloys).

The depletion of dislocations & the high stresses necessary for nucleation of new ones have been blamed for high flow stresses & significant strain hardening in  $Ni_3Al$  during superplastic deformation.

#### **4. Conclusion**

In this chapter, we have demonstrated the concepts of creep and superplasticity. Moreover, we discussed the factors affecting the creep, effect of stress and temperature on the creep curve, creep mechanism of crystalline material, creep resistant material, and creep in nanomaterials in detail. Creep is defined as permanent distortion (plastic deformation) of a material over time under a constant load or stress. Moreover, we have illustrated the superplastic materials, which shows the extremely large tensile elongation before failure. Furthermore, we have outlined the superplasticity mechanism in polycrystalline material, and in nanomaterials. Thus, this chapter will provide the understanding to design new materials which are used at high temperatures like components of gas turbines, nuclear reactors, furnaces, rockets and missiles, etc.

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