# High temperature

High temperature is a relative term

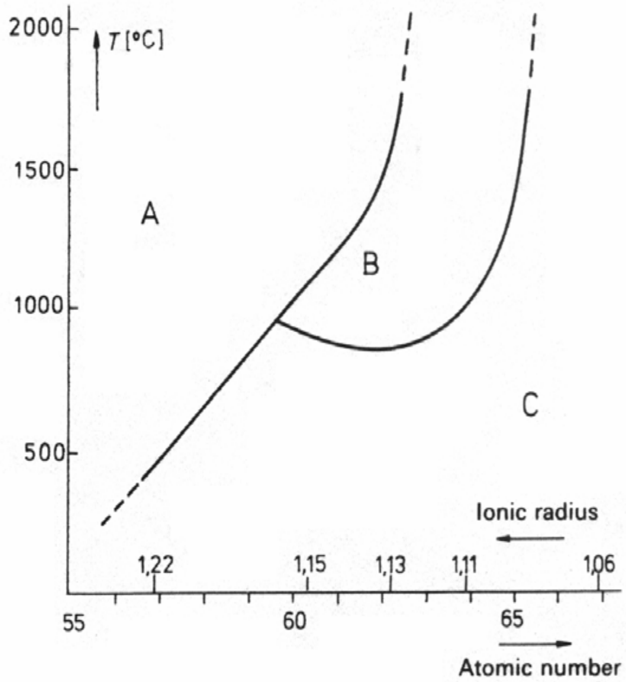



Fig. 1. Phase diagram for trivalent lanthanoid oxides, after *Goldschmidt*. For the A-, B-, and C-types, see text.

We use high temperature in order to reach stability areas of new phases and to increase reaction rates by increasing the mobility of atoms, ions and species…

Problems related to instrumentation and equipment increases exponentially with temperature

# Methods

***Mechanical processes***

Adiabatic compression (>$10^3$ °C)
Adiabatic shock waves (>$10^4$ °C)

**Resistance heating**

| | |
|---|---|
| Cr/Ni | (1100 °C) |
| SiC | (1500 °C) |
| Pt/Rh | (1700 °C) |
| $MOSi_2$ | (1800 °C) |
| $MO(H_2)$ | (2000 °C) |
| C | (2500 °C) |
| $ZrO_2$ | (2500 °C) |
| $MO_2 + M_2O_3$ | (2000 °C) |

**Electron-beam heating**

Focal spot fusion
Drip fusion (>3000 °C)

*Thermochemical processes*

**Flames**

$2H_2 + O_2$ (2660 °C)
$C_2H_2 + O_2$ (3100 °C)
$C_2H_2 + N_2O$ (>3100 °C)
Metal powder/$O_2$ flames
"skating sun"
(≈2500—4500 °C)

**Metallothermy**

Metal oxides, sulphides, and halides + Al, Mg, Ca, Zr, *etc.*
(≈2000—2500 °C)

*Electrical processes*

**Induction heating**

Inductive heating of electrically conducting substances
Crucible-free fusion
Open plasma torch (>$10^4$ °C)
Closed plasma torch (thermal disequilibrium)
(≈$10^3$—$10^4$ °C)

**Electric arc**

Arc fusion
(>3000 °C)
Electric-arc plasma torch
(>$10^4$ °C)
Arc-transport process
(≈2000—4000 °C)
Artificial suns
(≈2300 °C)

*Natural heat sources*

Solar furnaces
(≈3500 °C)

**Laser**

High-power $CO_2$ laser (>4000 °C)

# Measuring temperature

| | |
|---|---|
| Oscillator | 150 °C |
| Liquid filled thermometer | 400 °C |
| Thin film thermometer | 500 °C |
| IR thermometer | ? °C |
| Thermocouple | 1500 °C |
| Optical pyrometri | 2500 °C |

# Electron beam

Ca. 100 kVA

Vacuum ca. $10^{-3}$ mbar

Protected cathode

Small scale in electron microscopes

Needs a conducting sample

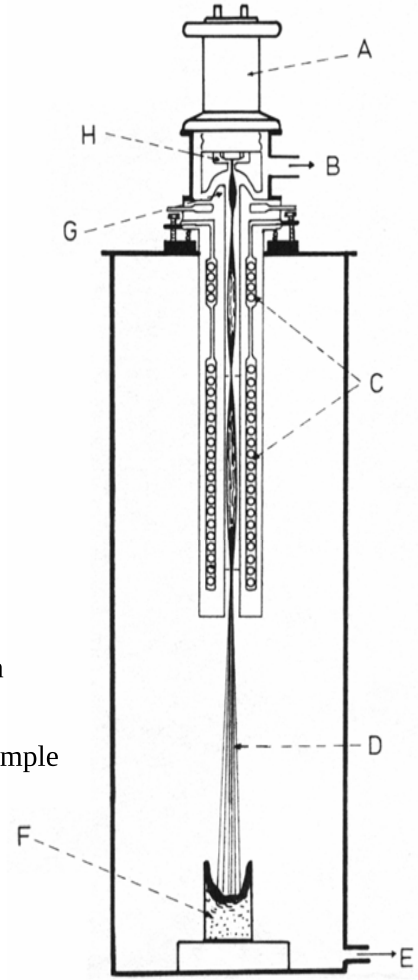



Fig. 2. Principle of an electron gun [25] for metal fusion in a field-free space. A: Insulator; B: pump for the electron gun; C: magnetic lenses; D: electron beam; E: pump for the preparation space; F: preparation; G: anode; H: cathode.

# Electric arc

Up to 7000 °C

A small distance between cathode and anode makes it possible to use low voltage

The heat is generated by a large current

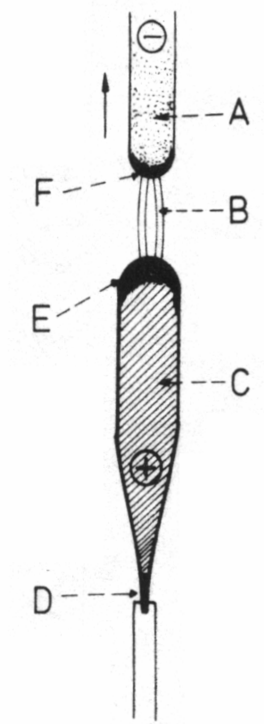



Fig. 3. Schematic representation of the arc transport process [30] for growing single crystals with transportation of the material in an arc. A: Feed electrode; B: arc; C: growing crystal; D: seed crystal; E: anode melt; F: cathode melt.

# Resistance heating

**Current is passed through a conducting material. Heat is generated by the electric resistance**

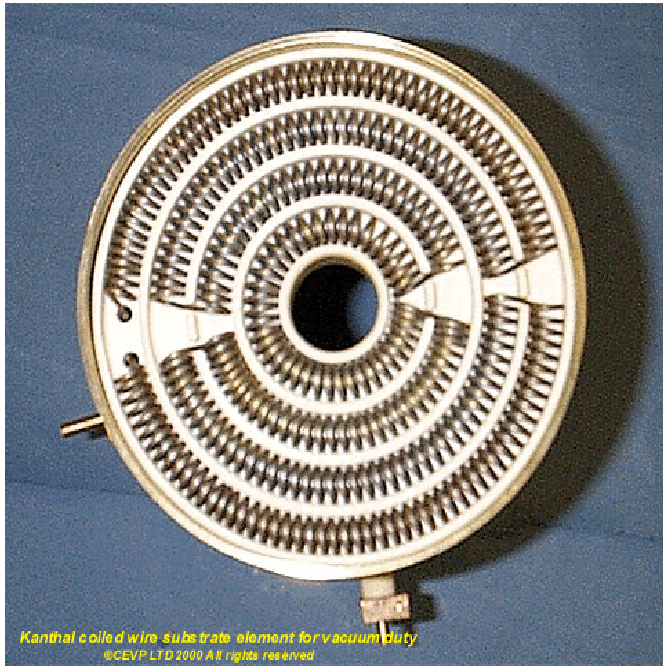
**The atmosphere is important**

**e.g. super kanthal to 1800˚C**

**Molybdenum**

**Graphite**

**May also use conducting oxides as heating elements (including transparent conducting oxides)**



# Induction heating

**By sending high frequence current (AC) through a copper coil, eddy currents are created in the enclosed (conducting) material, which will heat up the material.**

**The copper coil is often a watercooled tube**

**May allow crucible-less heating of metals.**

# Sun-furnaces

**Based on focused light.**

**Surprisingly stable temperature (2700°C, 1 hour, ± 10 °C)**

**The sample may be in vacuum or inert gas**

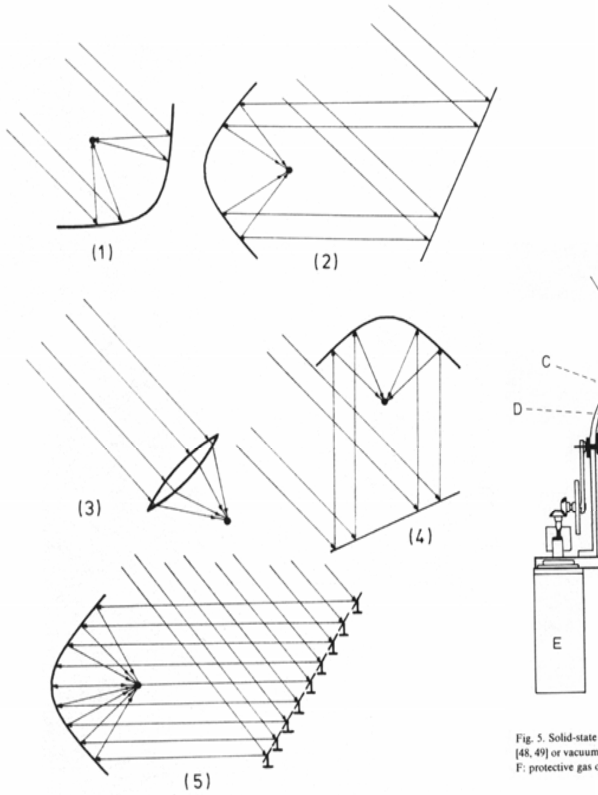



Fig. 4. Types of solar furnaces: (1) direct solar furnace; (2) horizontal heliostat; (3) type with a single lens; (4) vertical heliostat; (5) large-scale horizontal heliostat with illumination of the parabolic mirror by several plane mirrors.

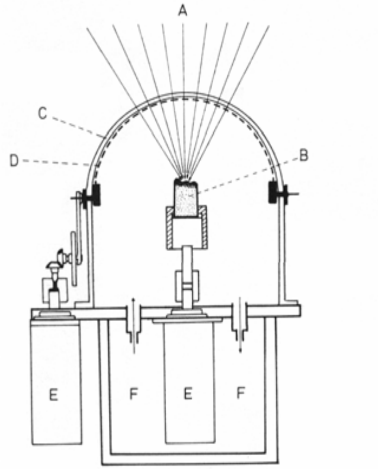



Fig. 5. Solid-state reaction, using a solar furnace and a specific gas atmosphere [48, 49] or vacuum. A: Sunlight; B: sample; C: quartz glass; D: stripper; E: motor; F: protective gas or vacuum.

# Artificial sun

**An arc or lamp may be used as a light source for focused light furnaces. The temperature is not as high as by using sun light**

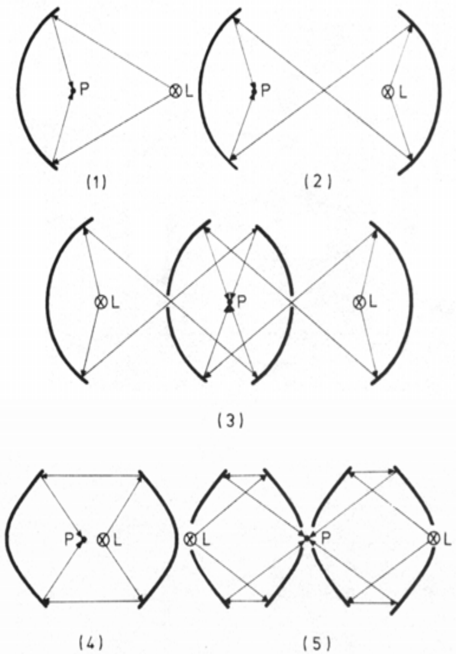



Fig. 6. Fundamental construction of optical heat sources (artificial suns). (1) Simple elliptical furnace; (2) double elliptical furnace; (3) multimirror system for two light sources with elliptical mirrors; (4) double mirror furnace with parabolic mirrors; (5) multimirror system for two light sources with parabolic mirrors (P = preparation; L = light source).

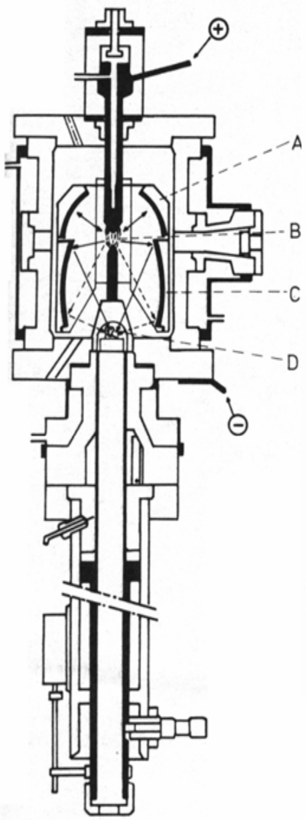



Fig. 7. Section through an electric arc furnace [52] for preparations under a specific gas atmosphere. A: Spherical mirror; B: electric arc; C: elliptical mirror; D: sample under a protective cap.

# Plasma torch

Temperatures of 8000 – 50 000°C

The plasma is produced by a strong electrical field across the gas, either arc or high frequency.

A fast gas stream prevents the current reaching the electrodes.

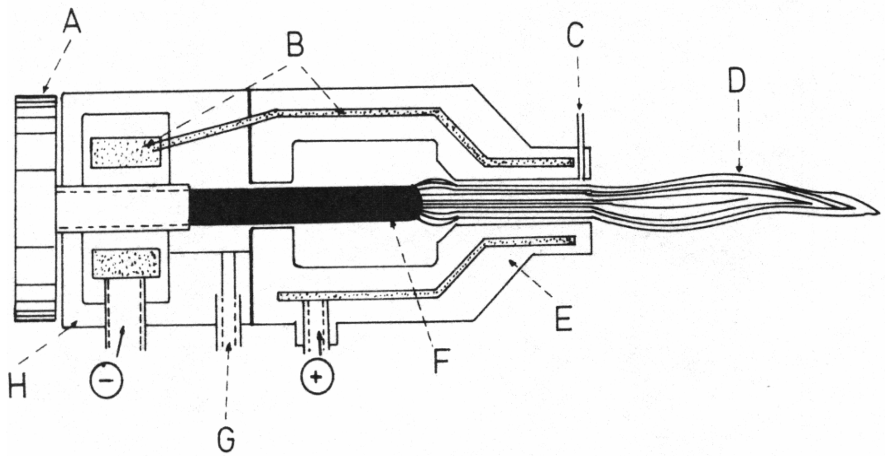



Fig. 10. Principle of an electric-arc plasma torch [78] for processing powdered materials. A: Adjustable electrode support; B: cooling; C: powder feed; D: plasma; E: anode; F: cathode; G: fuel gas supply; H: insulator.

# Plasma II

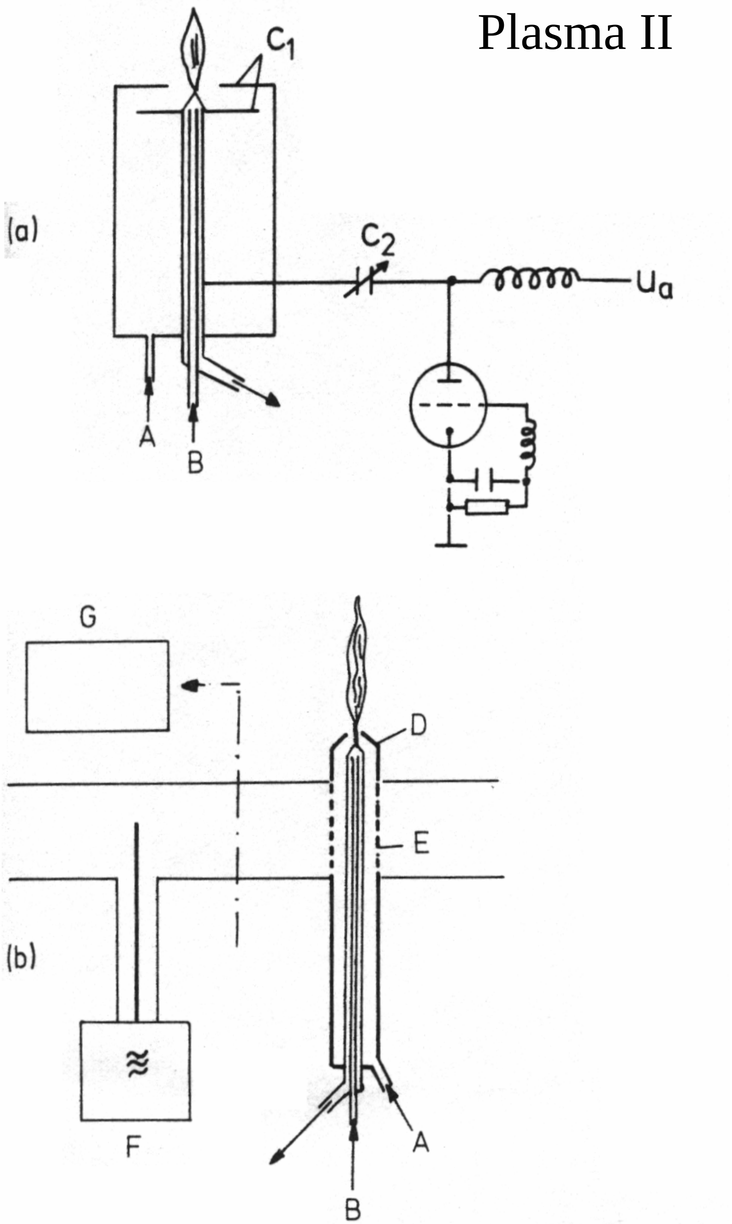



Fig. 12. Mode of operation of high frequency open plasma torches [92
several hundred MHz, (b) for 2400 MHz. A: Fuel gas; B: cooling; $C_1$, $C_2$:
tators; D: nozzle; E: electrode; F: HF generator; G: cross section.

# Plasma III

It is possible to create plasma in vacuum systems (0.1 – 1 mbar)

Use RF for producing the plasma

Often used to activate complexes.

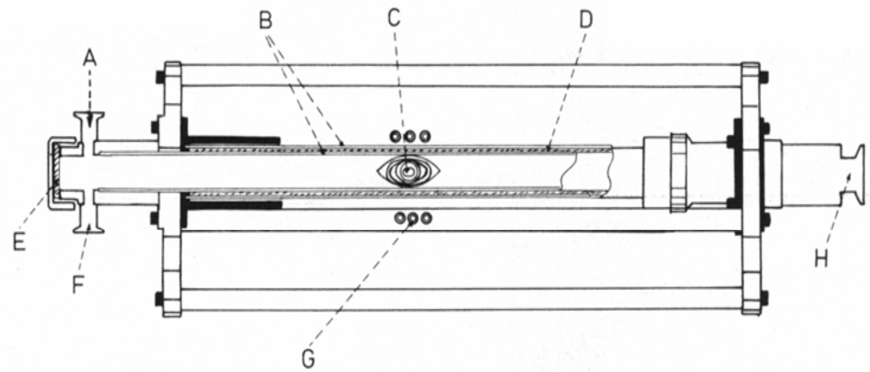



Fig. 13. Principle of a closed (low-pressure) plasma torch [100, 101] with inductive coupling of the HF energy. The torch can be displaced in a horizontal direction. The induction coil is fixed. A: Gas inlet (plasma gas); B: quartz tube; C: plasma; D: water cooling; E: window; F: pressure control; G: introduction coil; H: vacuum.

# Laser

The material should absorb radiation corresponding to the wavelength of the laser in order to create heat. ($CO_2$ laser: 10.6 μm)

Often used in thin film methods (ablation).

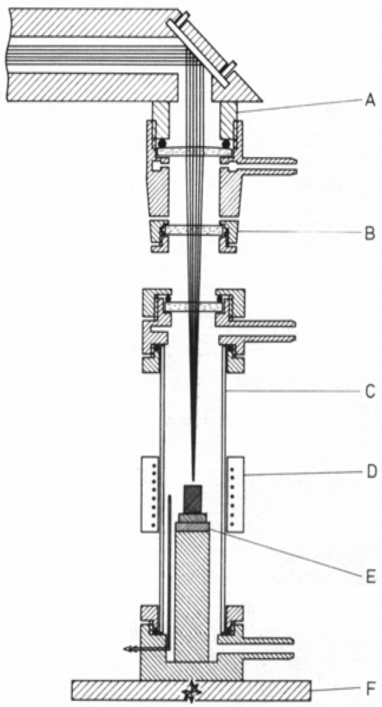



Fig. 14. Schematic representation of the performance of a high-temperature reaction with $CO_2$ laser energy under a specific atmosphere [121]. A: Collimator and focussing unit; B: protective window; C: sample space with the protective gas atmosphere; D: resistance heating; E: sample carrier; F: rotary, cross-slide and lifting stage.

# Crucibles

Inert metal, Ni, Pt, Ir

Alumina

Zirkonia

**Possible interdiffusion with crucible material**

Copper cooled crucible

Centrifuged crucible

Crucible-less melting (air stream, levitation)

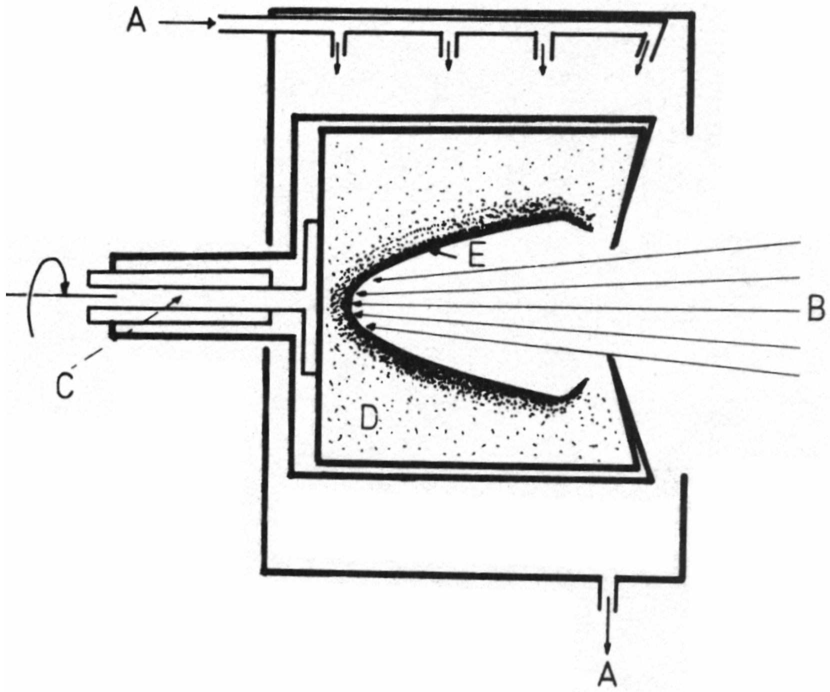



Fig. 9. Principle of the high-temperature centrifuge technique [73] for the prevention of wall reactions ("self-crucible method") in the heating of solids with radiant energy. A: Cooling; B: sunlight; C: centrifuge axis; D: solid phase; E: liquid phase.

# Microwave

**May be used for e.g. inorganic chalchogenides etc.**

**Metal must be crushed to small particles**

**Very fast heating (10 – 500 °C/min )**

**Used also for hydrothermal reactions and decomposition**