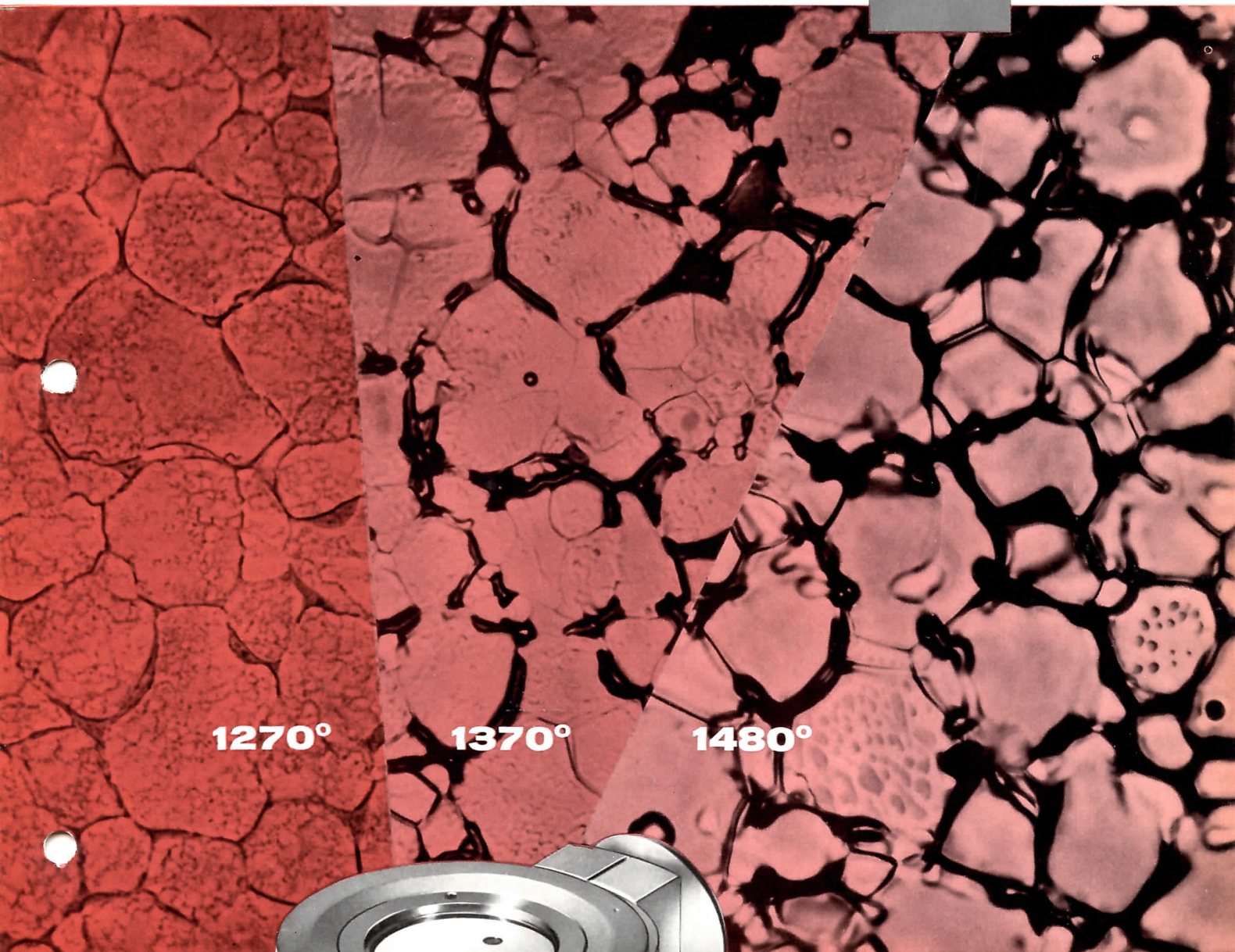


# Microscope Heating Stage 1750

*Leitz*  
WETZLAR

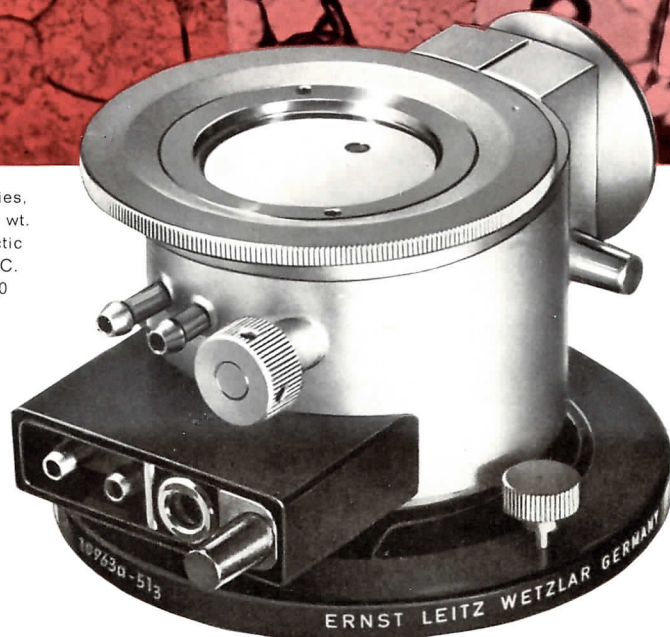


1270°

1370°

1480°

At the grain boundaries, rhodium and 1.5% by wt. silicon form an eutectic which melts at 1365 °C. Objective H 32 x / 0.60 Vacuum  $2 \times 10^{-5}$  Torr.



Examination in a vacuum or protective atmosphere of materials of any desired consistency. Shape and size of the samples can be modified to a large extent. Good contrast, as only a quartz window is situated between sample and objective. Freely suspended heating elements without ceramic part, thus low thermal capacity and quick temperature regulation.

ERNST LEITZ G M B H W E T Z L A R

515-68 b / Engl.

## Microscope heating stage 1750 for high-temperature microscopy

If the surfaces of cold polished metal specimens or sinter materials are examined under a microscope, the associations between melting, setting and diffusion, the growth of nuclei and the migration of grain boundaries can only be surmised from indications. Longer tempering or quicker quenching might have produced a structure of greater hardness or have resulted in higher elasticity or lower magnetization. As long as the sample is unseen during all the essential phases, however, questions of this kind can only be answered after long series of tests with systematic tempering programs. The desirability of direct observation is even more urgent if materials for crucibles and hot conductors, jets and turbines recrystallize under operating conditions, absorb or corrode alloy elements. Just such problems convince the metallographer, the ceramist and the process technologist of the importance of high-temperature microscopy. In the new microscope heating stage 1750 we have endeavoured to meet the essential requirements of the technologist: flexibility in test conditions. It is equally possible to study recrystallization of a red-hot wire, to melt crucible samples in a vacuum or to sinter powder in a stream of hydrogen. The description "Microscope heating stage 1750" simply means that this figure represents the upper limit of a simple measurement of temperature with commercial types of thermocouples. In principle higher temperatures can also be attained, but then the temperature of the sample has to be measured by pyrometric means. (see also page 6).



Fig. 1  
Surface of a sintered tablet of carbonyl iron powder tempered in hydrogen. Initial temperature 20 °C (68 °F).



Fig. 2  
After heating (1 hr.) to 1380 °C a closed surface has formed.

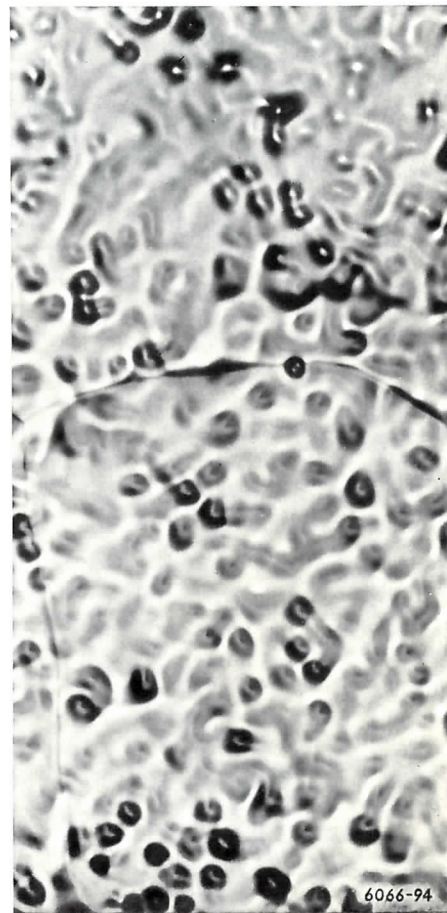


Fig. 3  
After a further 10 min. at 1380 °C thermal corrosion occurs; the  $\gamma$  grain boundaries become conspicuous.

# Technical description

## Design and operating principle

The microscope heating stage is designed for observation in incident light. It is especially suitable for use with our METALLUX® or PANPHOT® microscopes and is attached to the rotating stage No. 223 on these instruments (fig. 5).

Fig. 6 shows an exploded view of the heating stage. The vacuum chamber is mounted on a spherical bearing fitted with magnets. In this way the surface of the sample in the heating stage can always be swiftly aligned parallel to the surface of the microscope stage. The actual sample is placed over the thermocouple rather like a thimble. The capillary tube of the thermocouple ensures that the sample is kept in its proper position during examination under the microscope and also acts as a form of heat insulation. The wires of the thermocouple leave the vacuum chamber by way of a small insulated flange in the centre of the baseplate.

As a rule the sample of material is heated indirectly by means of low-voltage heating elements of tantalum. Screening plates, likewise of tantalum, enclose the "heating cylinder and concentrate the heat rays on to the sample. The heating elements and screening plates can easily be replaced by parts made of some other material, (see the section on "sample and heating"). Similarly, the entire heating stage can be dismantled into its component parts without difficulty for cleaning purposes.

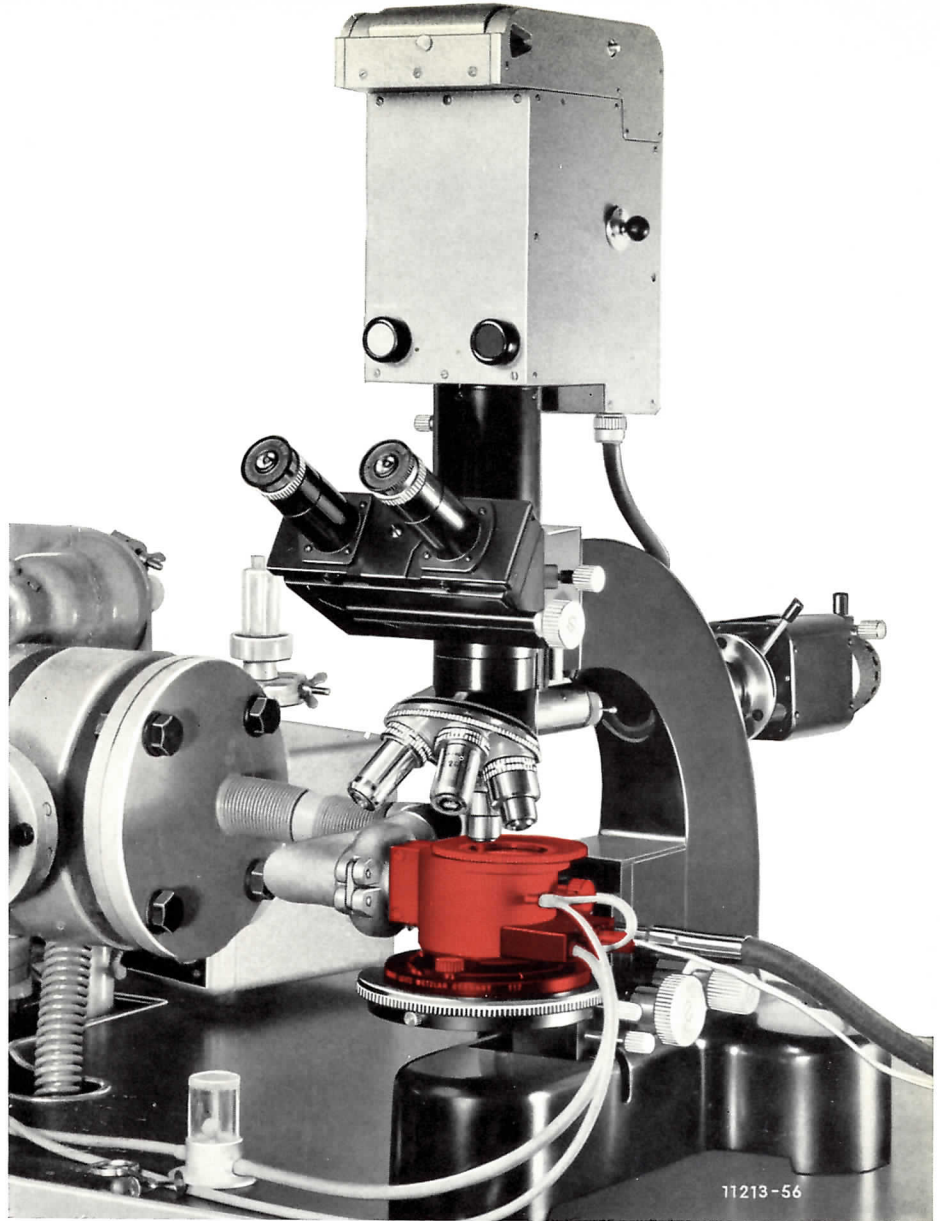


Fig. 5  
METALLUX metallurgical microscope with microscope heating stage 1750 (ready for operation) and fully automatic ORTHOMAT® camera for photomicrography.

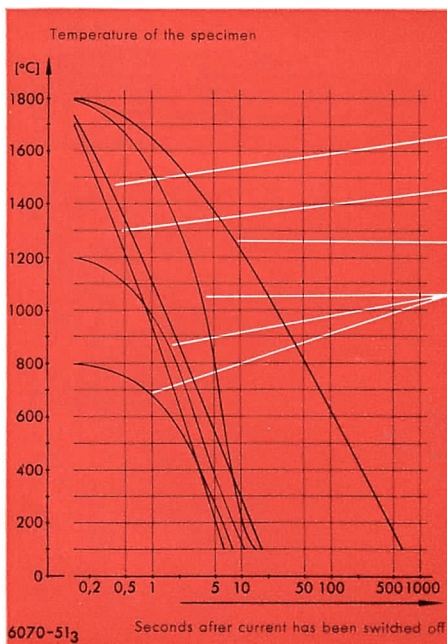


Fig. 4  
Temperature-time curves when cooling samples in a microscope heating stage 1750.  
Examples:  
Starting temperatures 1800 °, 1200 °, 800 °C.

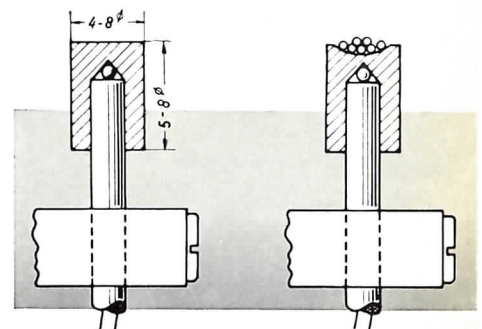


Fig. 7  
Fig. 8  
Samples and special crucibles are heated by radiation or contact from the surrounding heating elements. Diameter of samples 3mm.

## Component parts

### Spherical bearing

The vacuum chamber rests together with its screw cup on the spherical bearing fitted with magnets. In this bearing the chamber can be inclined up to an angle of  $10^\circ$  towards the optical axis and be fixed firmly in position. Simple and swift adjustment of this kind is important if the surface warps, sinters or melts locally at high temperatures.

### Vacuum chamber

The double-walled vacuum chamber is connected to the pump by means of the flange on the large vacuum joint at the side. When using gas flushing only this joint is closed by a cover. Inlet and outlet connections are provided in the outer jacket for the cooling water.

### Cover and quartz window

The heating elements and interior of the chamber are covered by a radiation protection plate. Only the surface of the sample remains visible through a small observation window. A quartz disc arranged eccentrically over the sample and rotatable through  $360^\circ$  closes the chamber so that it is vacuum-sealed and proof against excess pressure. If the window space above the sample has been clouded, it can be replaced up to 25 times by a clear part by turning the cover. This is particularly important if pyrometric measurements have to be taken at high temperatures.

### Objectives

The heating stage objectives are corrected to the thickness of the quartz glass window of 1.8mm. The following objectives are available: 5 x/0.09, 10 x/0.18, H 20 x/0.40, H 32 x/0.60, which can be used in conjunction with the incident light illuminator.

### Thermocouples

For examinations at  $1750^\circ\text{C}$  the microscope heating stage is equipped with a platinum-rhodium thermocouple which is officially suitable for temperatures up to  $1800^\circ\text{C}$  (E. L. 18 Heraeus or Pt-Rh 18 Degussa). For measurements up to  $1600^\circ\text{C}$  it is advisable to use a special platinum-rhodium thermocouple. The thermocouples can be quickly changed if necessary and can be repaired in the laboratory.

For observations at temperatures above  $1750^\circ\text{C}$  a micro-pyrometer can be used for measurements.

### Sample and heating

The fundamental idea behind the develop-

ment of the heating stage was to give the observer all possible freedom for his testing conditions. Figs. 7 and 8 are only examples of how the samples may appear. Fig. 10 shows how powder samples and grains can be heated up in a small receptacle of tantalum. Further shapes are shown in figs. 11 and 12. Heating elements and crucibles can be obtained from us. It is also possible to make similar or suitably adapted heating elements. The raw materials for this purpose (tantalum, megapyr, platinum etc.) are supplied by Messrs. Heraeus, Hanau or Degussa, Hanau. The basic outfit includes 2 heating elements of tantalum for indirect heating (fig. 9) and 1 heating element each of the types shown in figs. 10, 11 and 12.

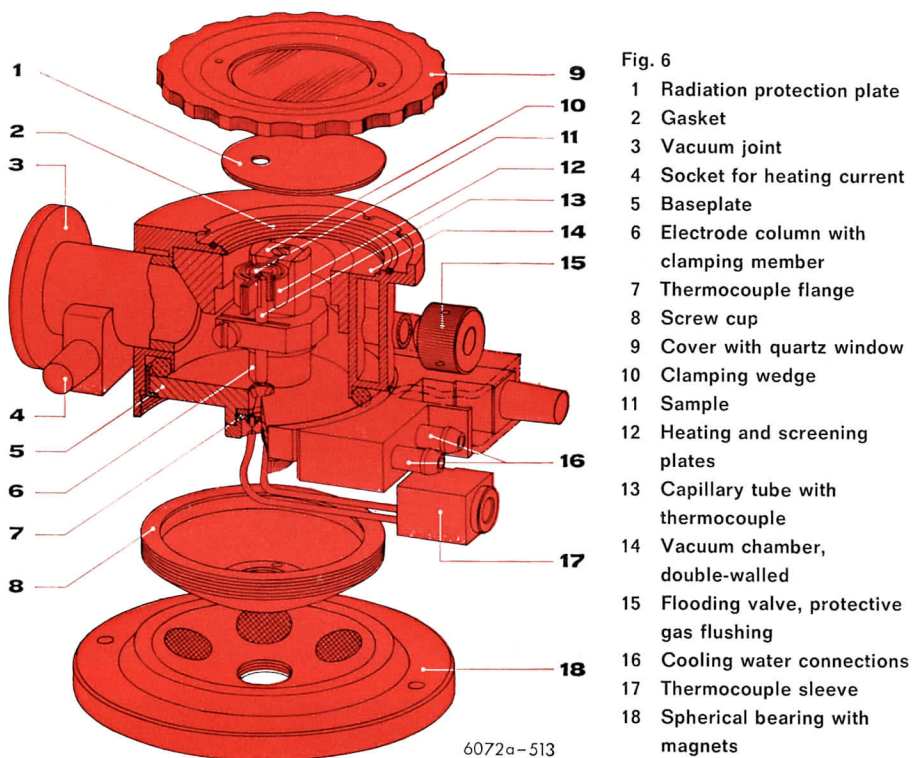


Fig. 6

- 1 Radiation protection plate
- 2 Gasket
- 3 Vacuum joint
- 4 Socket for heating current
- 5 Baseplate
- 6 Electrode column with clamping member
- 7 Thermocouple flange
- 8 Screw cup
- 9 Cover with quartz window
- 10 Clamping wedge
- 11 Sample
- 12 Heating and screening plates
- 13 Capillary tube with thermocouple
- 14 Vacuum chamber, double-walled
- 15 Flooding valve, protective gas flushing
- 16 Cooling water connections
- 17 Thermocouple sleeve
- 18 Spherical bearing with magnets

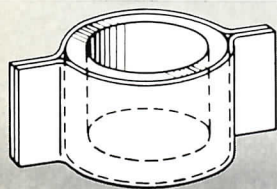


Fig. 9  
Tantalum bands  
for indirect heating.



Fig. 10  
Heating receptacles of tantalum, molybdenum, platinum etc. for small samples and sintered tablets. Sheet and wire samples are heated directly.

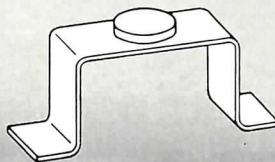


Fig. 11

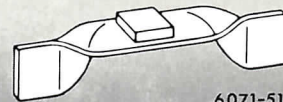


Fig. 12

6071-513



Fig. 13

Graphitization on white solidified rough casting under protective gas (argon). Objective H 32 x / 0.60, microscope camera ORTHOMAT

20 °C

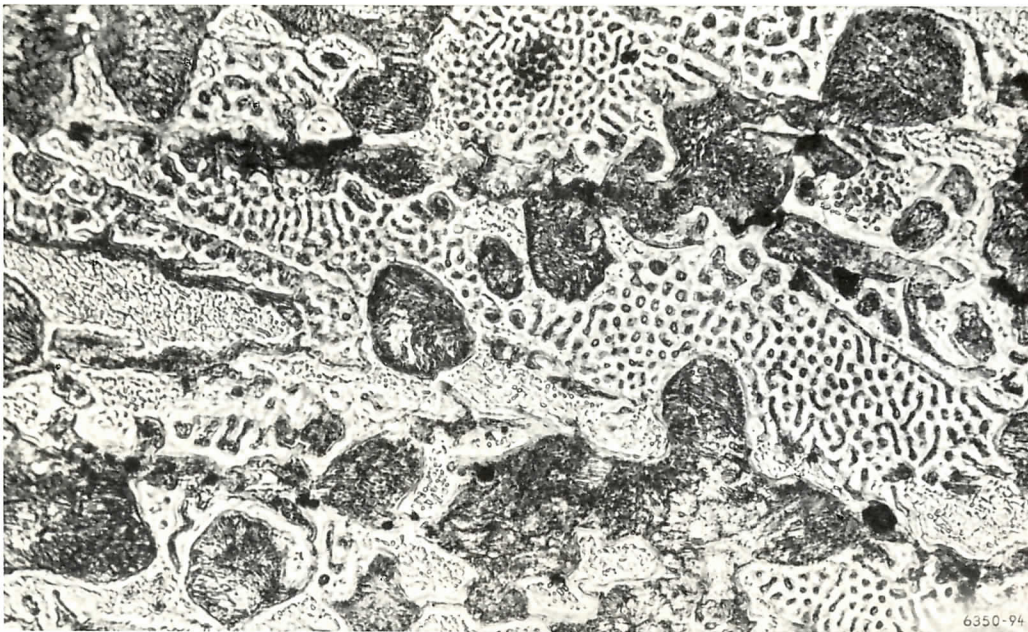


Fig. 14

950 °C

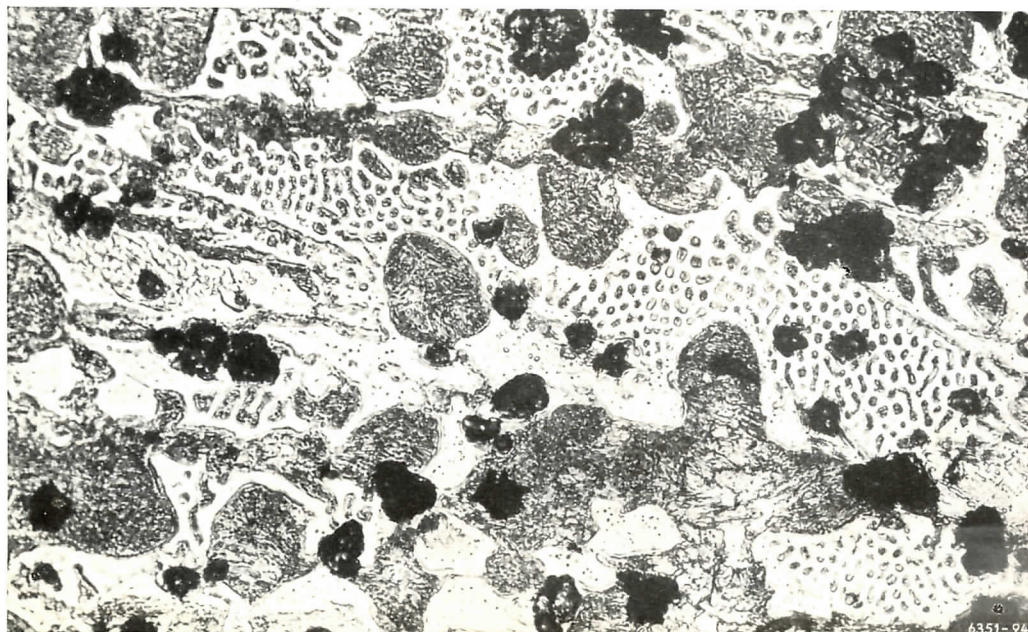
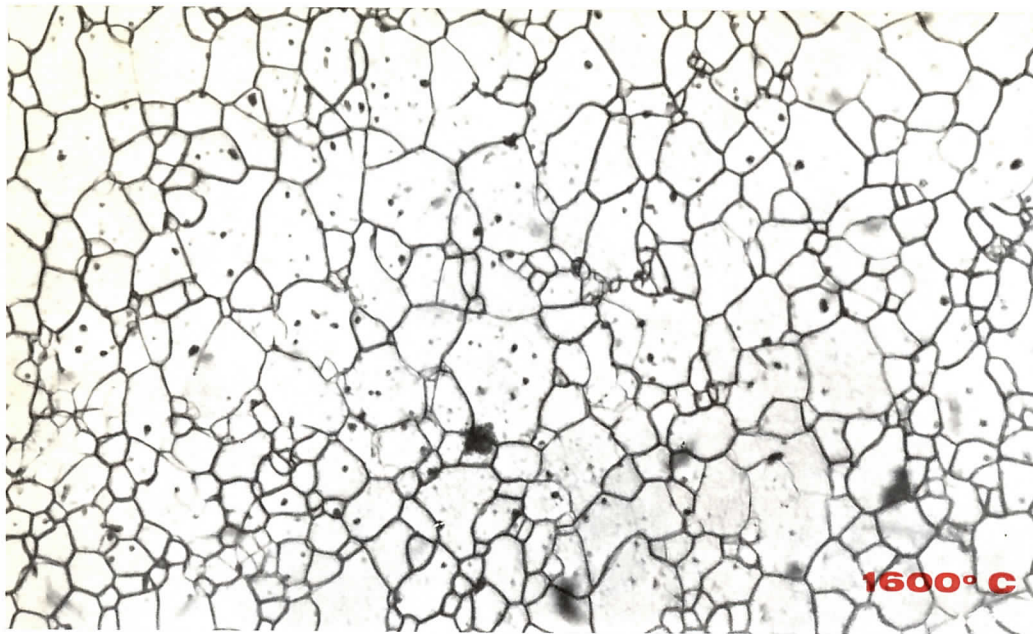
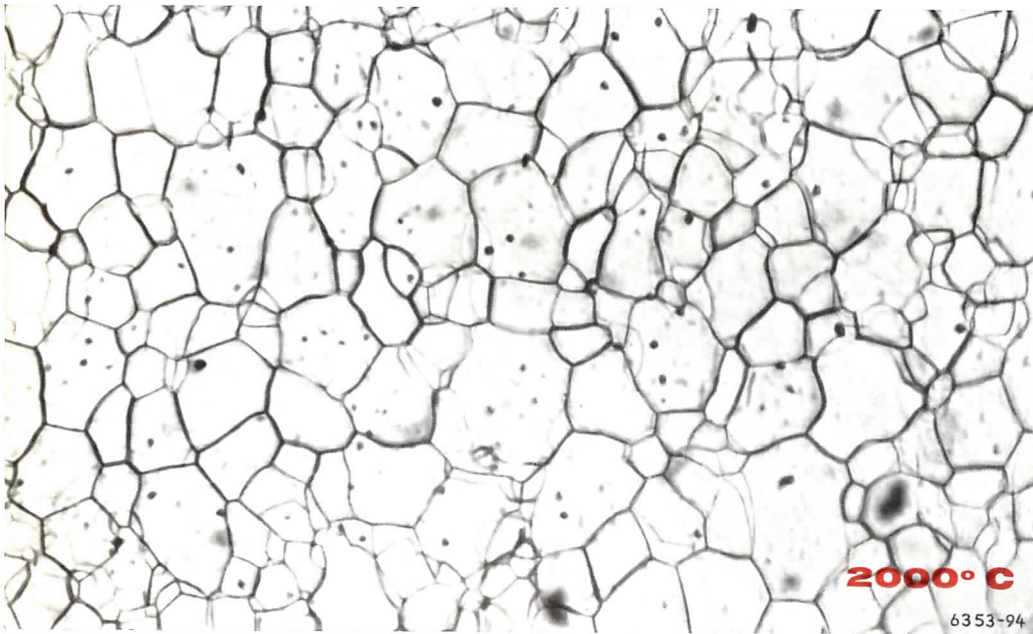


Fig. 15

950 °C after 20 min

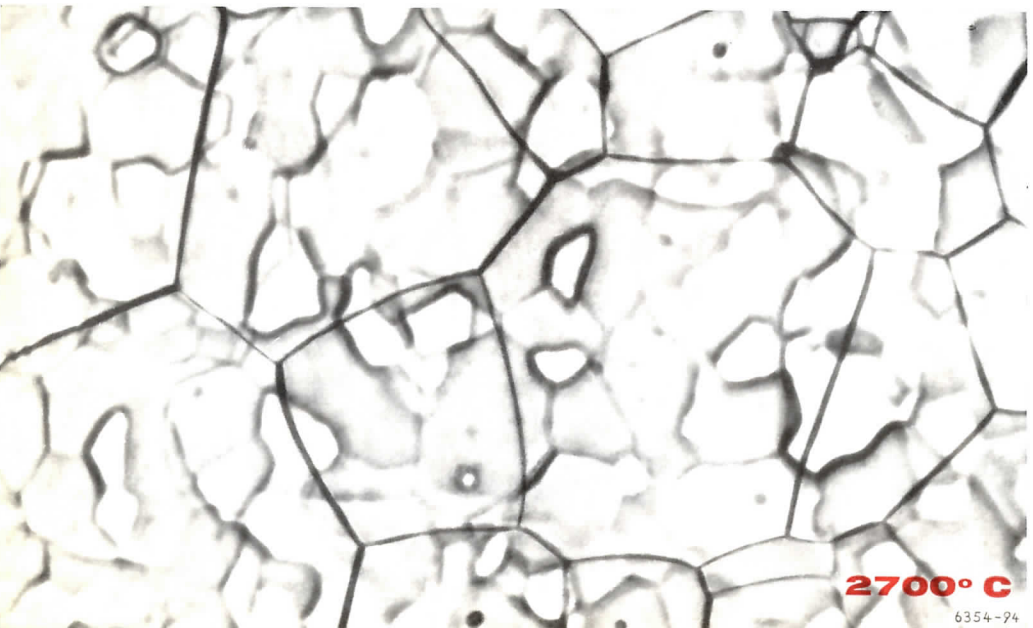


1600° C



2000° C

6353-94



2700° C

6354-94

**Use at extreme temperatures above 2000° C**

Under certain conditions even substances most difficult to melt above 2000 °C can be investigated with the microscope heating stage. Since commercially available thermocouples cannot be used above 1800 °C, measurements must be carried out with micropyrometers. An XBO 150 W Xenon burner combined with our Lamp Housing 250 is recommended as light source, particularly for photomicrography.



Figs. 16 - 18  
Tungsten at 1600 °C, 2000 °C, 2700 °C.

Recrystallization of tungsten plate at 1600 °C, 2000 °C, 2700 °C. The intense light emission of the sample itself was over-compensated by the powerful Xenon burner and blue filter 450m $\mu$ . As a result, pictures rich in contrast were obtained even at the most extreme temperatures.

Objective H 20 x/0.40. Lamp Housing 250 with XBO 150 W ORTHOMAT microscope camera.

Design subject to alteration without notice.

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