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RESEARCH MEMORANDUM

A SPECIALLY CONSTRUCTED METALLOGRAPH FOR USE AT ELEVATED TEMPERATURES

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NATIONAL ADVISORY COMMITTEE
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SUMMARY

A metallographic microscope was developed with provision for heating a specimen to 1800° F in protective atmospheres, that is, vacuum or gas. A special objective was constructed of reflecting elements with an unusually long working distance (7/16 in.) and a high numerical aperture (0.5). Changes in specimen microstructure were observed and recorded on 35-millimeter motion-picture film. The resulting pictures were projected as motion pictures and individual frames were cut and enlargements made for close observation. Structural changes upon heating a 0.35-percent annealed carbon steel and a 5-percent tin phosphor bronze specimen were observed and recorded. Newly formed microstructures were revealed by selective vacuum etching and specimen relief resulting from recrystallization and varying grain orientation.

INTRODUCTION

It has long been considered desirable (reference 1) to observe and to record changes in the microstructure of metals and alloys encountered at elevated temperatures. Motion pictures of the structural changes accompanying changes of temperature in the alloys are particularly desirable. Existing metallographic equipment is unsuitable for this purpose and must be modified in the following ways: The specimen must be enclosed in a furnace (heater plus protective chamber) with a controlled temperature and atmosphere, and the objective must give good resolution at an unusually long working distance.

A metallographic microscope was developed at the NACA Lewis laboratory which has a stage furnace for heating and enclosing the specimen in a protective atmosphere, a special microscope objective with large working distance and numerical aperture, and a motion-picture camera. The metallographic microscope is described herein and its use illustrated in observing structural changes in two specimens at temperatures up to 1650° F.

INSTRUMENT DESIGN AND CONSTRUCTION

The table and the optical bench of a standard metallurgical microscope are used to mount the components of the elevated-temperature metallurgical microscope (fig. 1). The furnace and the objective are mounted on one base at a distance from the light source, camera, and eyepiece mounted on another base. The vacuum system is placed at the rear of the table.

Furnace

The furnace (fig. 2) is designed to operate at temperatures up to 1800° F, to have a minimum heat loss, and to be of a minimum size. It is also necessary to keep the emissivity of all heated parts that "face" the chamber wall to a minimum and to keep the vapor pressure of all heated furnace parts low.

The heater element (0.008-in. diam. cleaned tungsten wire) is wound in a helical groove on a steatite core. Steatite is used because it can be readily machined when "soft" and then fired at a relatively low temperature (2100° F) to obtain a high density and low porosity. If specimen temperatures in excess of 1800° F are desired, an aluminum oxide core can be substituted to increase core rigidity. The tungsten wire is wound at a temperature of 800° F to give an increase in ductility, thus reducing the amount of springback. This particular heater element gives excellent performance and long life.

A platinum cylinder is fitted over the heater core. This cylinder serves as a specimen holder, as a means of conducting heat to the specimen, and as a radiation shield to minimize the energy loss to the chamber wall. The specimen (1/8-in. diam. and 1/2-in. long) is inserted in a hole bored transverse to the platinum cylinder near the top. The specimen is secured by a set screw on the axis of the cylinder. Heat is radiated to the platinum cylinder from the heater element and conducted to the specimen. Platinum is used because the emissivity and the vapor pressure are low and the thermal conductivity is high.

The specimen temperature is controlled by means of a continuously variable autotransformer and is measured by means of a thermocouple and potentiometer. This standard platinum - platinum plus 13-percent rhodium thermocouple is spot-welded to the end of the specimen, as shown in figure 2.

The heater element, platinum cylinder, platinum radiation shield, and specimen are enclosed by a stainless-steel chamber. A 1-millimeter-thick fused quartz furnace window is inserted into the wall of the chamber between the specimen and the microscope objective.

Rim cooling is provided for the window by water flowing through a circular channel in the window mounting. Cooling is also provided at the bottom flange of the chamber to prevent overheating of the neoprene gasket seal, thus reducing outgassing.

Protective Atmosphere

The specimen must be heated in vacuum (0.1 micron of mercury or less) or in a protective atmosphere to prevent corrosion of the polished surface. A positive-displacement rotary-type mechanical pump with a free-air capacity of 100 liters per minute is used as a roughing pump. A 2-inch-diameter, fractionating, organic vapor-diffusion pump with a capacity of 60 liters per second at 0.1 micron of mercury pressure is used with the mechanical pump to obtain pressures less than 0.1 micron in the system. A hot filament-type ionization gage is used to measure the pressure (fig. 3). A Pirani gage is used to check the roughing procedure and to measure higher pressures when a gas is introduced into the system. No attempt has been made to measure the pressure at the specimen location. Required pressures at the gage locations were determined by performance.

If the vapor pressure of some constituent in the specimen is excessive, it may be possible to control the rate of sublimation by introducing an inert gas into the chamber, thus increasing the chamber pressure. Provision is therefore made for introducing gas through the furnace base. If etching of the specimen is required, a gas that will selectively attack the specimen at the operating temperature may be introduced in controlled amounts.

Optical Design

The first decision made in the optical design was to place the specimen alone within the furnace and all optics outside (fig. 3). The specimen is observed through a window placed in the wall of the furnace chamber. The working distance of conventional objectives of 0.5 numerical aperture or greater is too small (approximately 1/16 in.) to permit their use with the previously mentioned furnace design, which requires a 7/16-inch distance from specimen to objective. In order to obtain this working distance, a reflecting-type objective consisting of a system of centered spherical surfaces was chosen as capable of being ultimately developed to give image quality fully equal to that of a conventional refracting objective. Reflecting objective designs reported on by Grey (references 2 to 5) emphasize the potentialities of this type of objective. In this instrument a 0.5 numerical-aperture objective is used as a conservative basic design capable of further improvements, such as a greater numerical aperture if desired.

The basic form of the objective consists of a fixed convex mirror (fig. 4) supported by a spider and an adjustable concave mirror. Adjustment of the separation of the two mirrors corrects marginal spherical aberration of the system and tilt of the concave mirror centers the system to obtain zero coma on the optical axis. Longitudinal chromatic aberration introduced by the furnace window is compensated for visible and ultraviolet light by the plano-convex lens.

Primary magnification of the objective was chosen as 100 in order to produce a theoretical image resolution of 0.002 inch on the film (specimen resolution, 0.00002 in.). The conservative resolution of 0.002 inch is intended to prevent any loss of photographic detail. Optimum visual magnification is 500 diameters as determined by the conventional 500 to 1000 times numerical aperture and by experiment. This is obtained by a 5-power eyepiece. Focusing is accomplished by prefocusing the eyepiece on cross hairs located the same distance from the specimen as the film plane in the camera and then using the standard focusing knobs and observing the result in the eyepiece.

Köhler-type vertical illumination is provided by imaging a zirconium arc light source on the convex mirror of the objective (fig. 4). The light transmitted from the source through the splitter plate is prevented from being reflected back to the camera image by means of a light trap. Reflected light from the plano surface of the plano-convex lens in the objective is diverted from the camera image by tilting the lens 1°. Aberration caused by the 1° tilt is innocuous over the field of view because of the large focal length and small aperture ratio of the lens. A light shield in the objective intercepts light reflected back from the cover glass toward the image.

General Construction

The reflector objective is mounted on a standard microscope body tube to utilize the focusing mechanism (fig. 1). A second microscope focusing mechanism is used to raise and to lower the objective with respect to the specimen, to scan the specimen, and to permit a preselected area to be followed during the expansion of the furnace components. The specimen and the furnace are moved horizontally by means of screws in the furnace base.

Inasmuch as structural changes in materials are to be studied and such phenomena are related to a time factor, it is desirable to view the resulting pictures as motion pictures to give an integrated appearance of structural changes. The 35-millimeter film is the largest size available in long rolls and for which projectors are available. In addition, a great variety of photographic emulsions are available in

the 35-millimeter size including color and those having high contrast and very high resolution. A standard 35-millimeter motion-picture camera is therefore used in the instrument with the lens and turret removed and the drive mechanism modified.

Exposures are controlled by an intervalometer. The camera shutter-release mechanism is actuated by a solenoid, which is energized at controlled intervals for predetermined periods of time by the intervalometer. The interval range is from 20 seconds to 9 minutes and the period can be varied from 1/5 second to 90 seconds. The correct exposure is determined by a null-balance-type exposure meter consisting of a 6-candlepower lamp that illuminates both a photocell and a white plastic target. The target is located at the image plane of the eyepiece (fig. 1). While observing the image and the adjacent target, the operator adjusts the voltage across the lamp until the two fields appear to be of equal brightness. The photocell current is then proportional to the illumination of the image. The colors of the image and of the target are somewhat different; however, the use of a blue-green filter over the eyepiece eliminates this variable.

INSTRUMENT EVALUATION

In order to evaluate the instrument as constructed, photomicrographs were made of two different alloys at elevated temperatures. A specimen of 0.35-percent-carbon steel, annealed 30 minutes at 1650° F and furnace cooled was polished and etched with 3-percent nital. It was then heated slowly to 1650° F in vacuum and the microstructure changes were observed and recorded. Figure 5(a) shows the rate of heating of this specimen in the furnace and the resulting photomicrographs of the metal structure made at various temperatures up to 1650° F. The pearlitic structure at room temperature before heating is shown in figure 5(b). The first change in specimen appearance occurred at approximately 800° F (fig. 5(c)) and was apparent on visual examination by a slight increase in surface relief. This figure shows an increase in definition, which is attributed to surface relief. The resolution obtained by the reflecting-type objective is evident by the clarity of the lamellar spacing. At about 1200° F, contrast of the lamellae in the pearlite aggregate became less. The breakdown of the lamellae continued as the temperature increased, and at about 1300° F a salt-and-pepper structure appeared, as evident in figure 5(d). As the temperature was increased to 1400° F, the spots appeared to coalesce and to grow. At 1500° F, austenite grain boundaries were observed in the former ferrite regions (fig. 5(e)). As the temperature increased to 1650° F, the spheroids tended to dissolve in the austenitic grains and the austenite grain boundaries become more prominent (fig. 5(f)). The volume change accompanying transformation increased the relief of the specimen surface considerably.

Most of the former pearlite boundary was retained in the structure at 1650° F and can probably be attributed to the relatively high vapor pressure that exists at grain boundaries (reference 6). These boundaries were etched rather deeply by this loss of material. With prolonged heating the austenite grain boundaries were etched in the same manner. The austenite grain boundaries cross this former pearlite boundary at several points. The pearlite boundaries observed at 1650° F apparently have no significance other than to mark the location of the pearlite that previously existed.

The observed structure changes in this steel agree with those anticipated; however, the temperature at which these changes occurred is higher than the iron-carbon equilibrium diagram would indicate. This discrepancy can be accounted for largely by two factors: The rate of heating of the specimen was much too great to give an equilibrium structure at any given temperature. Also, a structure may have to exist for a sufficient length of time to be vacuum etched before it is apparent.

Photomicrographs were not made on cooling of this specimen because the surface relief was too great. After two transformations (α-γ and γ-α) it was impossible to find an area flat enough to be focused sharply. Also, the structure becomes quite complicated with three sets of grain boundaries, that is, the original, the austenite, and the final transformation structure. It may be feasible to observe and to record the transformation resulting from cooling from the austenite structure. The specimen should not be etched prior to heating in vacuum, the rate of heating should be high, and the time at maximum temperature should be short. It is thought that this technique may result in a structure with less relief and confusion.

A specimen of 5-percent tin phosphor bronze (1/2 hard), was polished and etched with potassium dichromate and then heated to 1500° F in vacuum. Figure 6(a) shows the heating cycle and the microstructure of this alloy at various temperatures from room temperature to 1500° F. The microstructure at room temperature (fig. 6(b)) showed the familiar strain lines and twinned structure of this bronze. At 800° F (fig. 6(c)) a number of individual strain lines became shorter and some disappeared, indicating a degree of stress relieving.

At temperatures of about 1100° F, considerable relief was observed. Such relief is probably due to one or more of the following factors:

(1) At a particular temperature stress relief occurred with an accompanying deformation.

(2) Grains that were highly strained probably recrystallized with an accompanying change in orientation.

(3) Adjacent grains having different orientation would expand differently when heated.

The specimen temperature was maintained at 1270° F for 2 hours. Figures 6(d) and 6(e) are photomicrographs made at the beginning and end of this period. Evidence of new or recrystallized grains appears in figure 6(d). The individual spots of spheroidal constituent decreased in size. A stain surrounding these spots originally was vaporized at this temperature. The structure at the end of 2 hours at 1270° F (fig. 6(e)) had a soft and diffused appearance primarily because of increased relief. At 1500° F (fig. 6(f)) the recrystallized structure was not readily apparent and the surface relief of the specimen increased.

DISCUSSION

Using vacuum as a protective atmosphere for a specimen results in a loss of alloy constituents having a high vapor pressure. Such a loss may be reduced by introducing a gas for protective atmosphere and increased pressure. In many instances, vacuum etching is beneficial, as it is selective in action and provides continuous etching of the specimen. When a new structure or constituent is formed, etching may be necessary to make it appear. This etching may be accomplished by holding the specimen at temperature in vacuum.

A specimen may be chemically etched prior to heating to make the microstructure visible and to permit the selection of a field that is representative of the specimen. After heating, vacuum etching may augment chemical etching and result in a deeply etched structure, which may represent the original microstructure. A newly formed microstructure existing at the observation temperature may be difficult to discern because of this deeply etched pattern. Such an effect should be considered when interpreting photomicrographs and perhaps controlled when observing changes in microstructure at elevated temperatures. This control can be effected by modifying experimental procedure.

Contrast in the image is an important factor in effective resolution of detail. This image contrast was reduced primarily by condensation of vapor from hot objects in the furnace to the inner surface of the furnace window and also by reflection of illuminating light from the furnace window and compensating lens. Deposits disappeared from the furnace window to some extent when the window became hot as the cooling-water flow rate was reduced.

In elevated-temperature photomicrography, it is desirable to use a fine-grained high-contrast photographic emulsion to enhance image contrast. The very small range of exposure of this type of emulsion made the exposure meter a necessity.

A first-surface mirror is used to reflect the image into the eyepiece tube. The mirror slides off the optical axis when the image is to be recorded on film. A thin, uncoated pellicle mirror would be advantageous in that it would permit observing and recording the image simultaneously while diverting only 8 percent of the available light to the eyepiece.

It also appears feasible to observe the microstructure of material under stress at elevated temperatures by using the optical system herein described with a stage furnace suitably equipped and instrumented. Such a technique would be valuable in studying the mechanism of creep.

CONCLUSIONS

From the design and construction of a special metallograph and a preliminary evaluation of its use at elevated temperatures, the following conclusions are drawn:

1. Photomicrographs of excellent resolution and motion pictures of changes in microstructures can be obtained at elevated temperatures.

2. The investigation indicates that the instrument will be valuable in the study of aging and recrystallization of high-temperature and refractory metals and alloys.

3. The objective constructed of reflecting elements produces excellent image quality and high resolution. This type of objective appears to be ideally suited for elevated-temperature metallography.

4. Microstructures can be revealed by selective vacuum etching and specimen relief resulting from recrystallization and varying grain orientation.

5. The specimen operating temperature may be increased from 1800° F to a higher temperature by substituting a more refractory material for the heater core.

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3. Grey, David S.: A New Series of Microscope Objectives: II. Preliminary Investigation of Catadioptric Schwarzschild Systems. Jour. Optical Soc. Am., vol. 39, no. 9, Sept. 1949, pp. 723-728.

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6. Chalmers, B., King, R., and Shuttleworth, R.: The Thermal Etching of Silver. Proc. Roy. Soc. (London), ser. A., vol. 193, no. A 1035, July 21, 1948, p. 479.

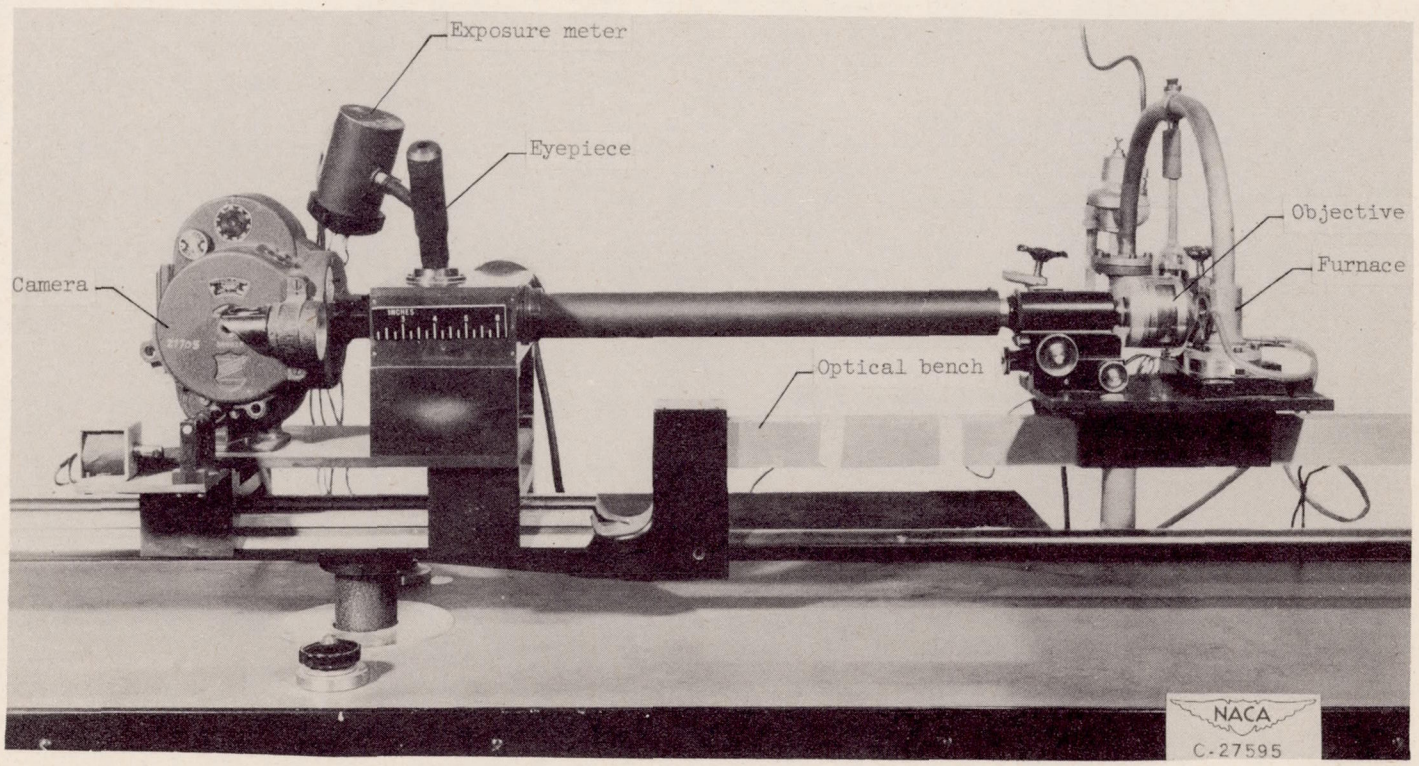


Figure 1. - Components of elevated-temperature metallograph.

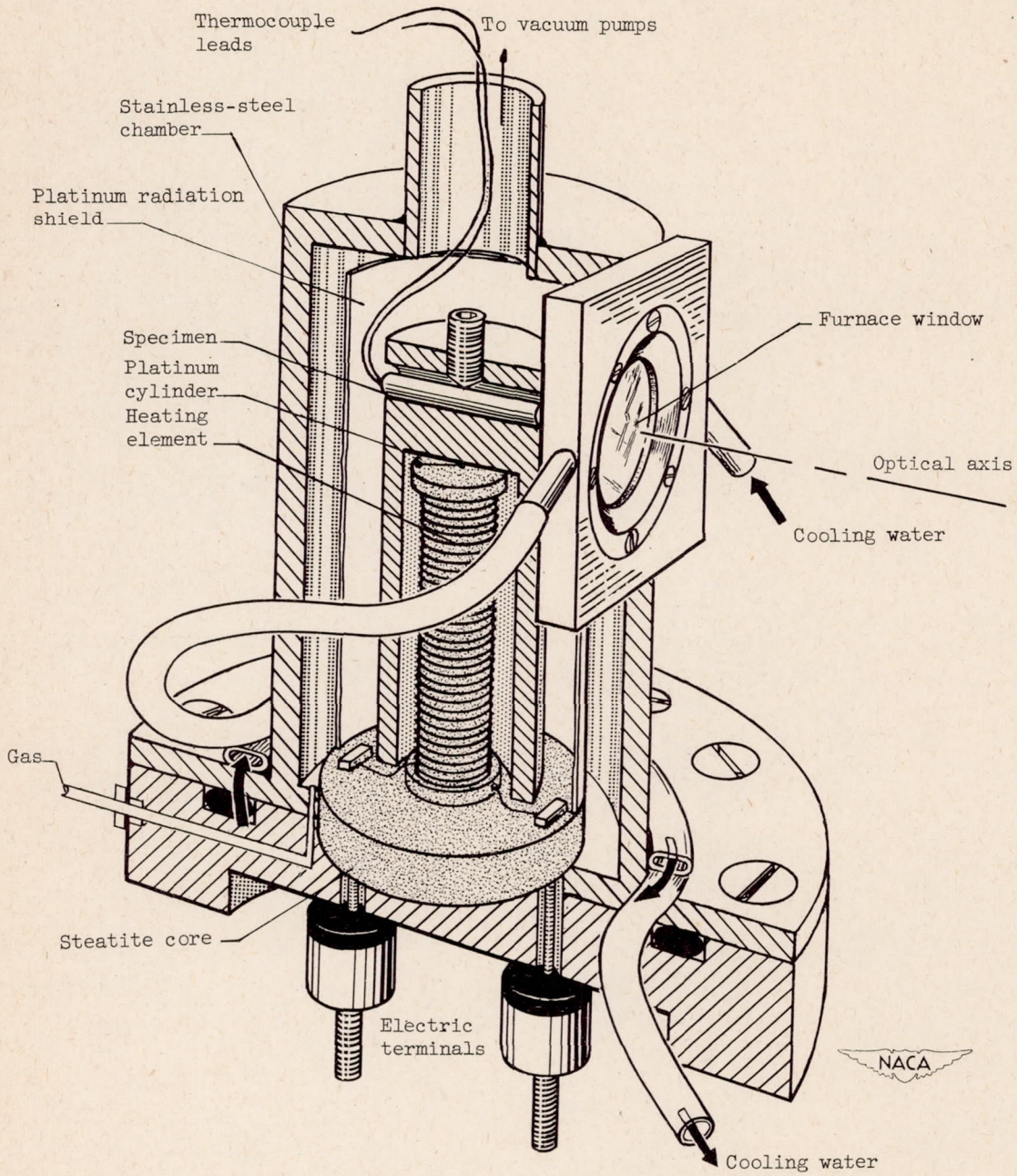


Figure 2. - Microscope furnace.

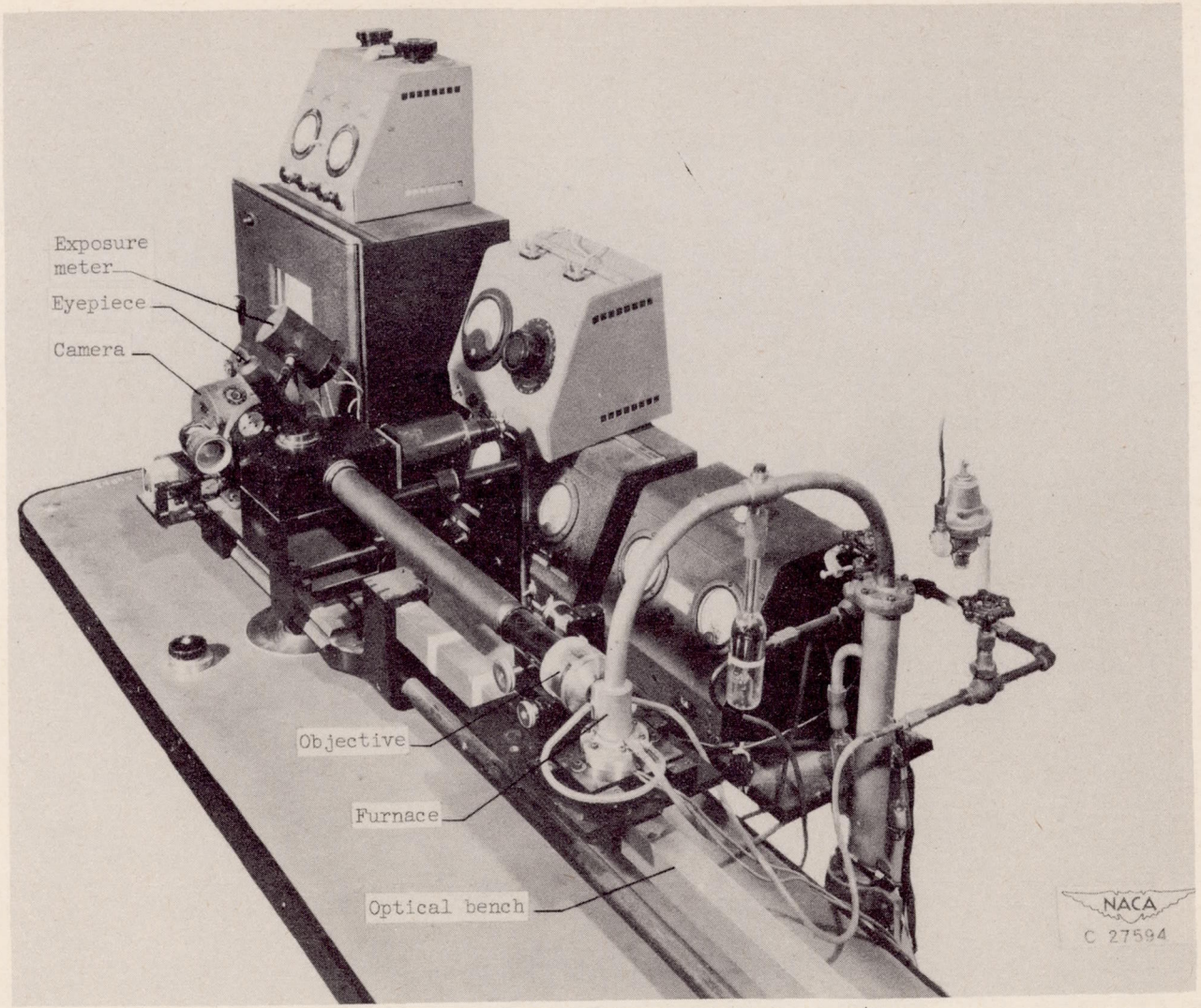
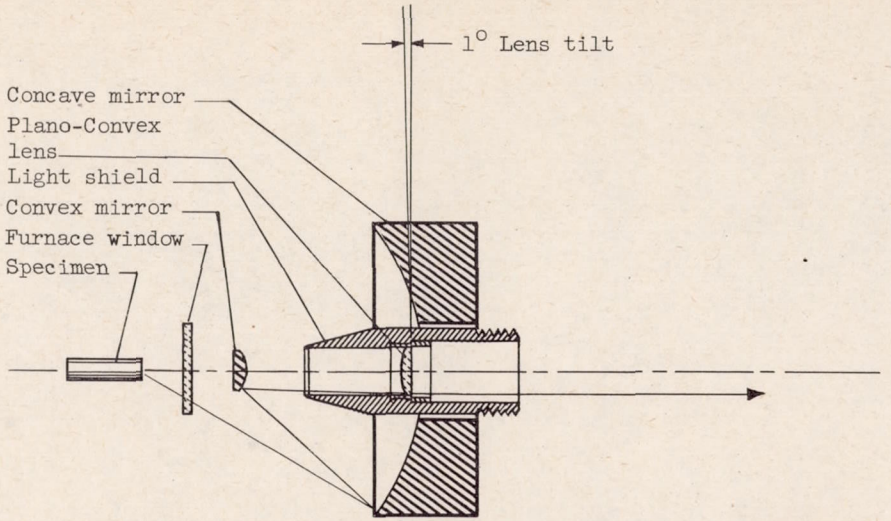


Figure 3. - Elevated-temperature metallograph.



Detail A - Full size section through objective.

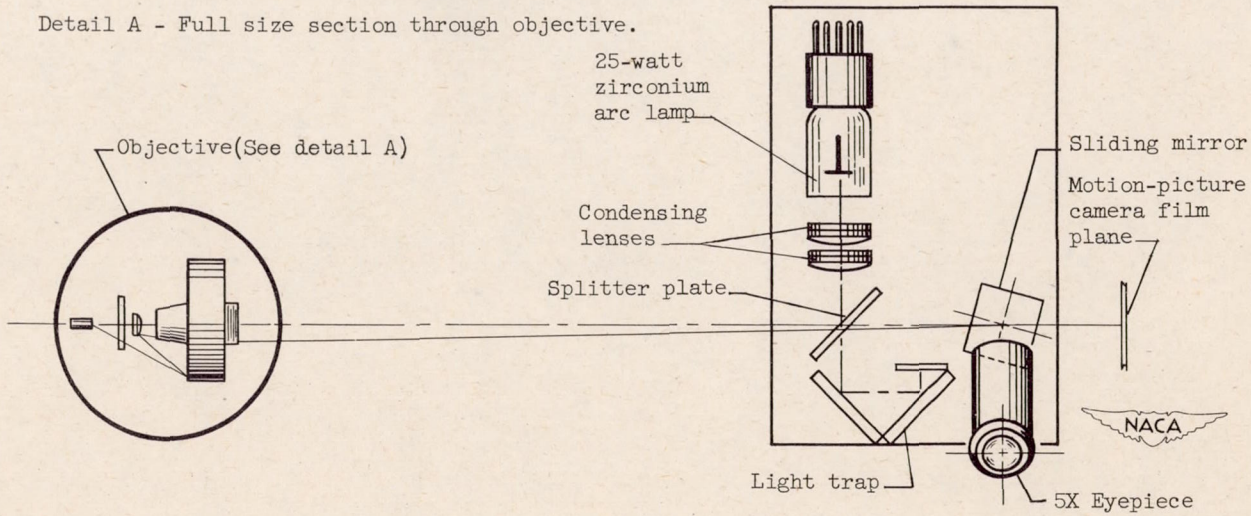
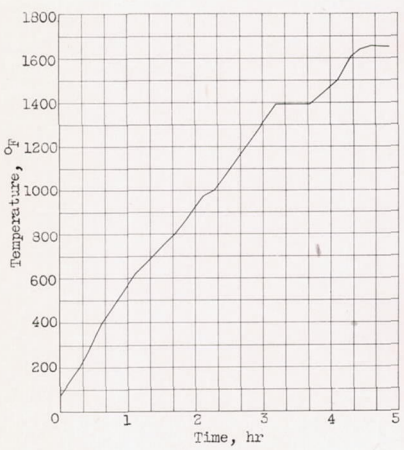
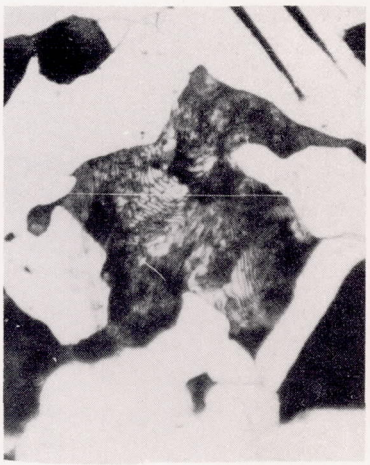


Figure 4. - Optical system.

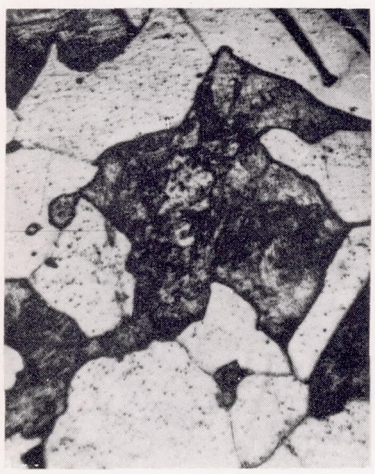


(a) Heating curve.

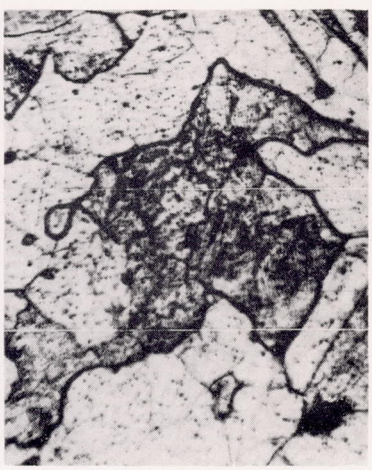


(b) Room temperature.

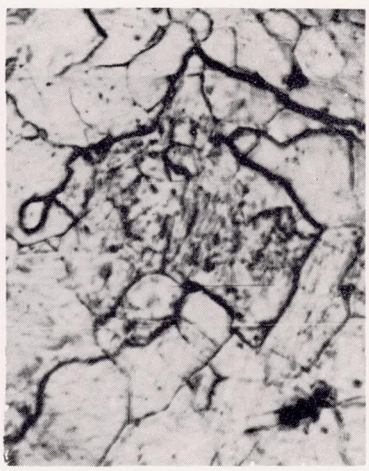
(c) 800° F.



(d) 1390° F spheroidization of carbides



(e) 1550° F alpha-gamma transformation.



(f) 1650° F solution of carbides.

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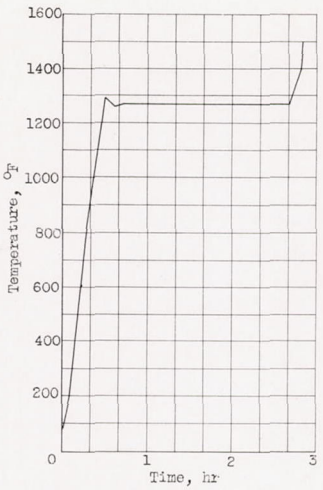
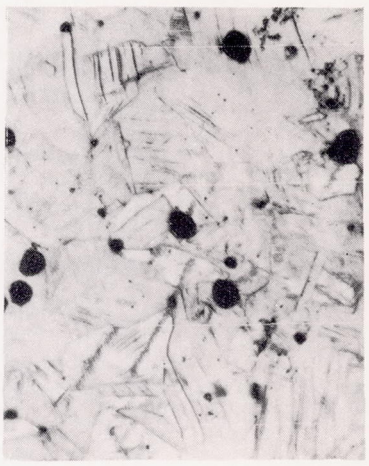
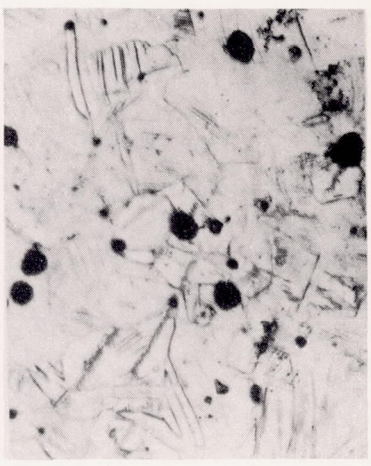
Figure 5. - Microstructures of 0.35-percent carbon steel observed at various temperatures reproduced from motion-picture negatives. X330.



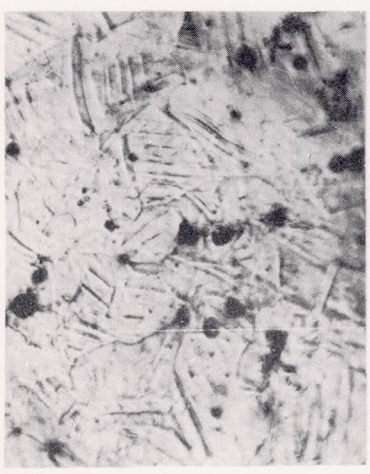
(a) Heating curve.



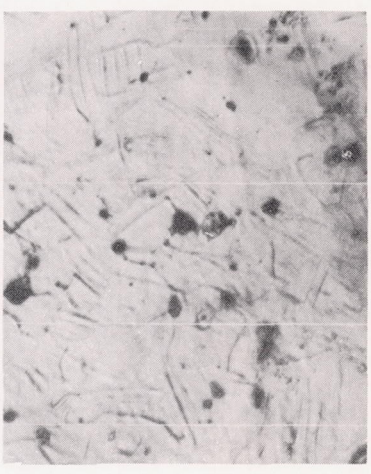
(b) Room temperature.



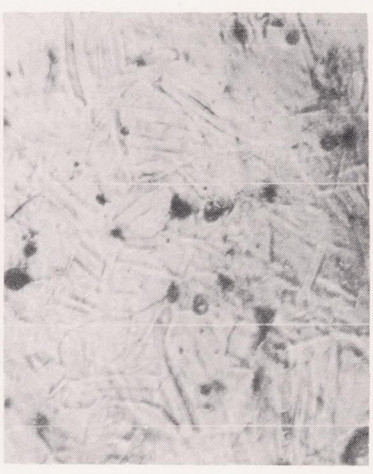
(c) 800° F.



(d) 1270° F.



(e) 1270° F after holding 2 hours.



(f) 1500° F.

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Figure 6. - Microstructures of 5-percent tin phosphor bronze observed at various temperatures reproduced from motion-picture negatives. X330.