

UNCLASSIFIED

AD 404 583

*Reproduced
by the*

DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION

CAMERON STATION, ALEXANDRIA, VIRGINIA



UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

63 3 4

D1-82-0240

CATALOGED BY ASTIA

AD No. ~~404583~~

Also available from the author

BOEING SCIENTIFIC
RESEARCH
LABORATORIES

The Microscopy of Metals in Transmitted
Ultra-violet Light

404 583

A. J. Forty

Solid State Physics Laboratory

March 1963

MAY 24 1963

D1-82-0240

The Microscopy of Metals in Transmitted Ultra-violet Light

by

A. J. Forty

March, 1963

Solid State Physics Laboratory
Boeing Scientific Research Laboratories

THE MICROSCOPY OF METALS IN TRANSMITTED ULTRA-VIOLET LIGHT

ABSTRACT

It is suggested that transmission ultra-violet light microscopy might provide a useful technique for metallurgical studies of the alkali metals and of silver and silver-based alloys. It is possible that this technique, providing the resolution of detail permissible with good optical microscopy for the examination of fairly thick metallic specimens, will fill the gap between transmission electron microscopy and X-ray diffraction topography for metallographic observation. Of the many potential applications the detection of microstructure by decorating techniques, the study of precipitation and other chemical reactions inside metals and the direct observation of phase changes are listed as those which are likely to be of immediate interest. The paper presents some results of a preliminary investigation of the microstructure in specimens of potassium, using impurity decoration and ultra-violet light microscopy, to demonstrate the experimental feasibility of these proposals.

- * This investigation was initiated by the author during a period of leave-of-absence from the University of Bristol when he was temporarily employed at The Boeing Scientific Research Laboratories, Seattle, U. S. A.

Although it has long been known that the alkali metals and, to a limited extent, silver are transparent to ultra-violet light (Wood 1918), there has been no attempt, hitherto, to exploit this remarkable phenomenon in metallurgical studies. Prompted by the intriguing possibility of looking inside a "transparent metal", the author has recently undertaken a survey of the experimental requirements and of the possible applications of such studies. This note lists some of these, and gives a preliminary account of microscope observations made with potassium in ultra-violet light to demonstrate their feasibility. A significant feature of the observations is the use of relatively thick specimens (films several microns in thickness) with the degree of magnification and resolution normally associated with optical microscopy. Transmission ultra-violet light microscopy might be expected, therefore, to provide an observational technique to fill the gap between high resolution electron microscopy (Hirsch 1959) and the more macroscopic technique of X-ray diffraction topography (Lang 1959).

Figure 1 shows the results of calculations by Briggs and Ives (1936) of the transmission of light in the near ultra-violet and visible part of the spectrum through films of potassium for several different thicknesses. Their calculations were based on measurements of the optical constants for potassium by a reflection method but the variation shown here can be verified, at least qualitatively, in transmission studies. This behaviour is typical of all the alkali metals, and clearly indicates the possibility of

transmission microscopy in ultra-violet light. The critical wavelength, λ_0 , at which the high transmission falls rapidly to the extinction normally associated with a metal is particularly significant. It has been described by Zener (1933) as the shortest wavelength at which the conduction electrons, behaving as a free electron gas, can be excited by the incident light; this is the wavelength corresponding to the so-called plasma frequency and is given by

$$\lambda_0 = \left(\frac{\pi m c^2}{N e^2} \right)^{\frac{1}{2}}$$

where m = mass of electron

e = charge carried by
electron

c = velocity of light

N = density of free electron

Zener's analysis, which accounts for the experimental observations surprisingly well, is used here as a basis for some of the proposals for the application of ultra-violet light microscopy.

The transparency of silver to ultra-violet light is thought to occur for different reasons. The variation with wavelength shown schematically in figure 2 for a thin film of this metal is based, again, on reported values of reflectivity. No direct

measurements of transmission have been made in this case, but the behaviour represented here has been confirmed in preliminary microscopic studies with evaporated films. The critical Zener wavelength for silver would lie in the far ultra-violet part of the spectrum and, like copper and gold, this metal would not be expected to show transparency to near ultra-violet light according to the free electron model. The "window" occurring at wavelengths around 3100 \AA can be explained in terms of the band structure of energy levels for the conduction electrons. The excitation of electrons to levels within the S-band accounts for the absorption of visible light, whilst inter-band transitions from levels in the S-band to levels in the P-band give rise to absorption in the far ultra-violet end. The window appears at those wavelengths for which photon energies are insufficient to excite the transition. In the cases of copper and gold the bands of levels overlap so that there can be no windows for these.

It is evident from figure 1 that, at least for potassium, and probably for all the alkali metals and silver, it should be possible to obtain transmission micrographs with specimens several microns in thickness if observations are made with light at wavelengths below the critical value. At this thickness the mechanical properties of foils are thought to become typical of those of bulk specimens and this is clearly an important consideration in metallurgical studies. The initial investigation described below shows that such observations are indeed possible.

It also illustrates an obvious application to examine the microstructure within the metal, by the decoration of grain boundaries and, possibly, sub-boundaries with precipitates of strongly absorbing impurity.

This idea of detecting a second phase by selective absorption suggests a further application in the direct study of precipitation reactions. Whilst reactions in systems based on the alkali metals and alloys of these are interesting, the precipitation of copper in silver/copper alloys is more useful as a metallurgical model. This experiment and a similar investigation of the internal oxidation reaction in silver/magnesium alloys are now in progress at Bristol. The microscope observations are made at a wavelength of 3100 \AA , with specimens in the form of thin foils, mounted on a heated stage so that the reactions can be followed directly.

The direct observation of the phase change at melting and of the martensitic structural transformation of the alkali metals at low temperatures are further interesting metallurgical applications of ultra-violet light microscopy. They are likely also to yield results of considerable physical significance, both in connection with the mechanisms of these processes and also in providing further information to relate the absorption of light with the electronic structure of these metals. By an extension of Zener's analysis we may expect the critical wavelength, λ_c , to

shift when an alkali metal undergoes a change of phase. For λ_c depends on N , the density of free electrons, which varies as the mass density, provided there is no change in electronic structure during the change. For example, in the case of potassium, where there is a decrease in density of between 2 and 3 per cent during melting, an increase of 30-40 Å in λ_c can be expected. It should therefore be possible to distinguish between molten and solid metal by making microscope observations at a wavelength close to λ_c for the solid. A molten zone should then be visible by bright contrast due to enhanced transmission. There is considerable interest in localised melting and in the reverse process of solidification. The direct study of a martensitic transformation, again using a possible shift in λ_c for detection, is equally interesting, though much more difficult to achieve experimentally.

Figures 3 and 4 illustrate that films of potassium at least one micron in thickness, are sufficiently transparent to ultra-violet light at wavelengths below λ_c (3100 Å) to permit transmission microscopy. These are ultra-violet transmission micrographs of parts of films of potassium containing a small addition of sodium. The films were prepared by distilling the molten metal into very thin evacuated cells, formed by sealing together two quartz plates in loose contact. The cells were heated to just above the melting point of potassium by immersion in hot water so that the metal could flow into the space between the plates. The films were solidified slowly by allowing the water to cool, or, more

rapidly, by the complete removal of the bath. Specimens prepared by this procedure are either single crystals or polycrystals containing only a few large grains. The microscope observations are made with light at a wavelength of 3050 \AA , using an arrangement consisting of a high pressure mercury lamp (having several strong spectral lines in the neighbourhood of λ_0), a grating monochromator having a large exit numerical aperture, a bench microscope fitted with aluminised mirrors and quartz refracting condenser and objective, an electronic image converter for visual observation and a film camera for photographic recording.

Figure 3 shows part of a grain boundary in one of the polycrystalline specimens. The boundary is heavily decorated by a strongly absorbing precipitation. Figure 4 shows the microstructure "visible" within a grain in another, similar specimen. These areas are totally absorbing for visible light and, therefore, are thought to be at least one micron in thickness (Wood 1918). The overall distribution of small, absorbing particles and the more regular arrangement of these in a form of network, apparent in figure 4, could be traced across the whole grain. The pattern is very similar to that of etch pits on the polished surface of a carefully annealed metal, and it is tentatively suggested that it represents an arrangement of sub-boundaries decorated by a finely dispersed precipitate. It has not been determined whether this precipitate is of pure sodium, an intermetallic compound or the product of localised chemical reaction with residual oxygen or

water vapour in the cell. Many similar observations have been recorded and will be described fully elsewhere. They are reported here in this preliminary manner to demonstrate the feasibility and potential usefulness of the technique of ultra-violet light transmission microscopy.

References

1. Briggs, H. B., Ives, H.E., 1936, J.O.S.A., 26, 238.
2. Hirsch, P. B., 1959, J. Inst. Met., 87, 406.
3. Lang, A. R., 1959, J. Appl. Phys., 30, 1748.
4. Wood, R. W., 1918, Phil. Mag., 38, 98.
5. Zener, C., 1933, Nature, 132, 968.

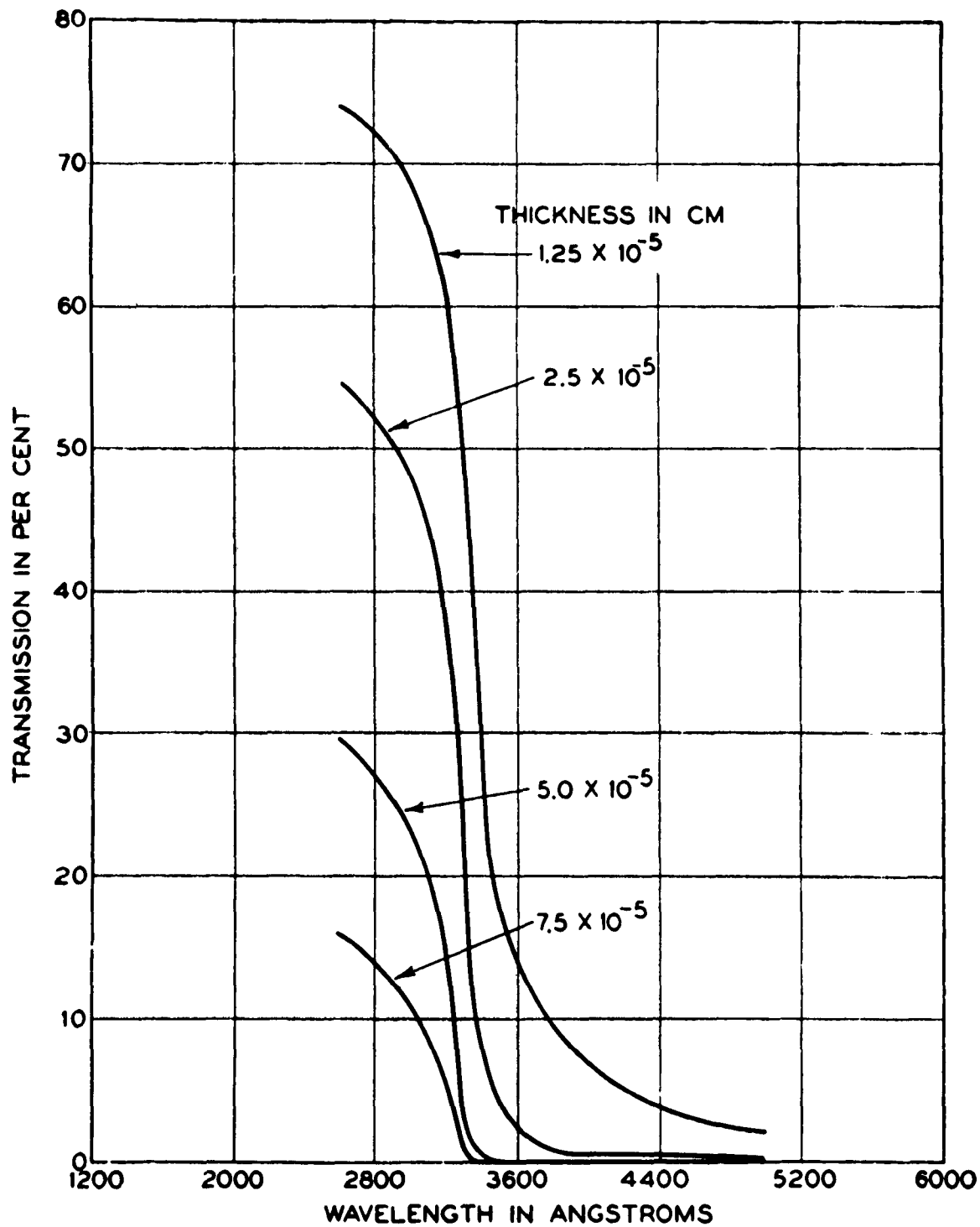


Fig. 1

Transmission curves for potassium in visible and ultra-violet light (after Briggs and Ives, 1936).

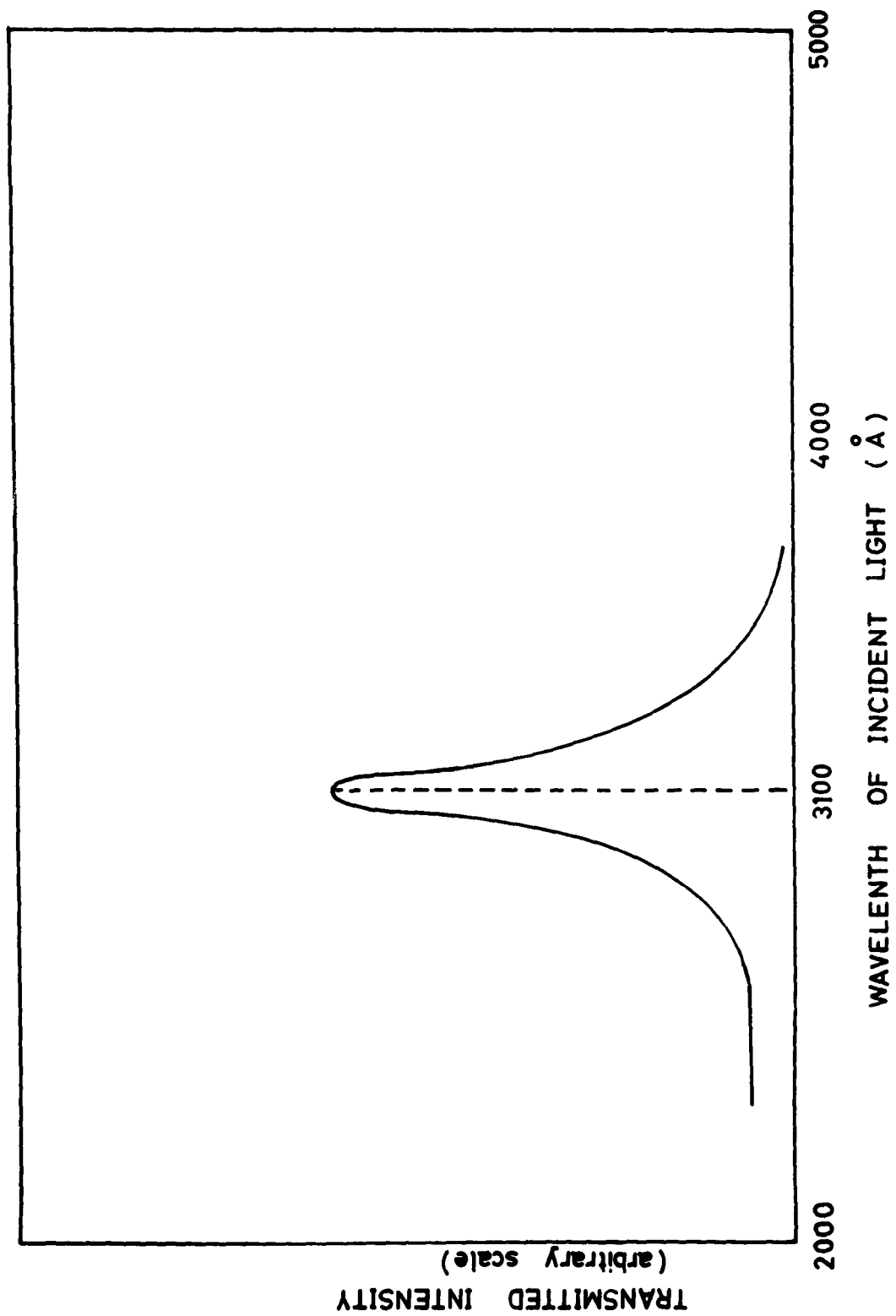


Fig. 2 Schematic representation of transmission for thin film of silver.

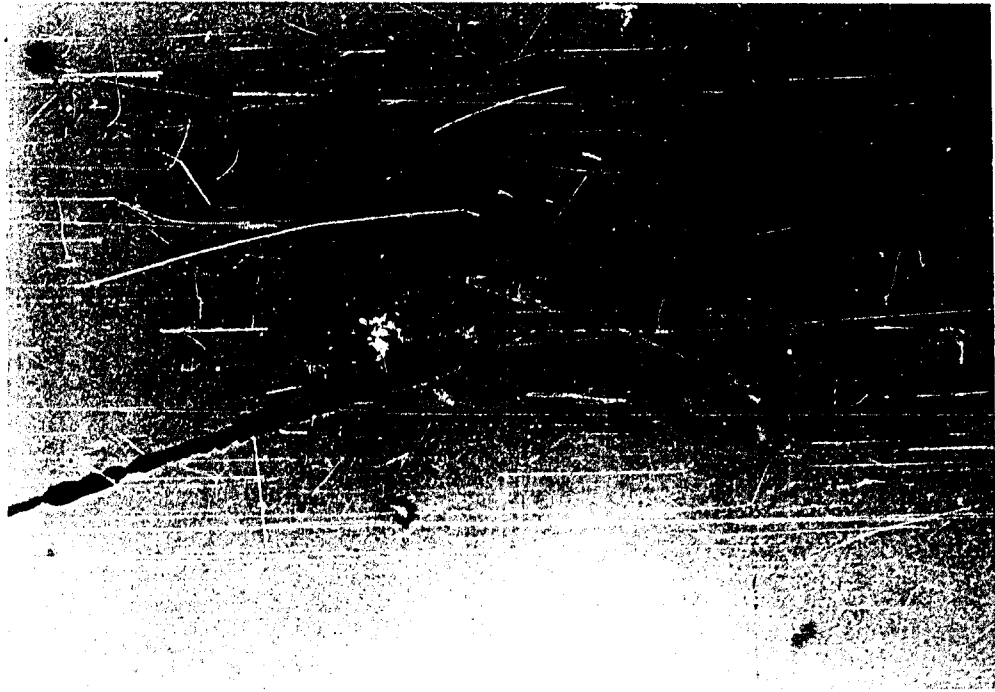


Fig. 3 Transmission ultra-violet light micrograph of part of polycrystalline sample of potassium, showing a grain boundary decorated by precipitation of impurity (X 1000)

