

THE
PHYSICAL REVIEW

TOTAL REFLECTION OF X-RAYS FROM NICKEL FILMS
PART II

BY HIRAM W. EDWARDS

ABSTRACT

Variation of critical angle with thickness of film for films deposited on platinum.
—The total reflection of x-rays ($\lambda=0.707\text{\AA}$) is obtained from a series of thin nickel films of thickness varying from zero to 2.05×10^{-6} cm. The films were sputtered upon thick platinum which was, in turn, sputtered upon a glass support. The critical angle for each film was measured. The values of the critical angle varied from 0.0040 radians (that of the bare platinum) to 0.0034 radians. With the exception of an increase in the critical angle (maximum value of 0.0043 radians) for the thinnest films, the critical angle decreased logarithmically with increasing thickness of the nickel film. The value obtained from the thickest film agrees with that calculated by the Lorentz dispersion formula. These results, together with those reported earlier seem to prove conclusively that the phenomenon of total reflection is not a purely surface phenomenon but is one which requires a layer of metal of probably definite thickness for the particular reflecting matter and wave-length of radiation used. An explanation for the variation of the critical angle with the thickness of the nickel films is given. This is based upon the assumption that the contribution to the intensity of the reflected ray made by the deepest electrons will be effective only when the total length of the path of the radiation in the metal is not too great. The maximum effective depth is that which reduces the intensity of the emerging radiation by absorption to a value less than that which may be detected in the reflected beam.

IN A previous report¹ the writer presented the results of an experimental determination of the critical angle of several thin nickel films sputtered upon glass supports. The results of that investigation gave values of the critical angles, for $\lambda=0.707\text{\AA}$, varying from a minimum value of 0.0016 radians, that of the unsputtered glass, to a maximum value of 0.0034 radians. The final value agreed with that calculated by the Lorentz dispersion formula, assuming a density of 8.75 gm/cc for nickel. The increasing value of the critical angle with increasing thickness of the nickel was taken as evidence for supposing that the electrons of the underlying glass support participated in the phenomenon of reflection and gave values for the critical angles which might have been construed as due to abnormally small values of the density of the nickel.

¹ Edwards, Phys. Rev. **32**, 712 (1928).

In order to obtain conclusive evidence to test this hypothesis, a second series of nickel films was prepared. The thicknesses increased from zero to a final value of about 2.05×10^{-5} cm. In this test, however, the nickel was sputtered upon thick platinum films. Eight platinum films were made by sputtering platinum upon flat pieces of glass. All the glass supports were cut from a single piece of glass and each received the same amount of platinum. Each platinum film was sputtered for 30 minutes under as nearly identical conditions of sputtering current, voltage, gas pressure, and cathode receiving-surface distance, as was possible to maintain. The thickness of the platinum was not measured but was thick enough to make the film entirely opaque to intense sources of light and to give the same value of the critical angle by x-ray measurements as was obtained from a film fully three times as thick.

The nickel films were sputtered upon the platinum mirrors under similar sputtering conditions except that the time of sputtering for the seven films varied from 2 to 50 minutes.

The critical angles were obtained by the method and with the apparatus described previously.² The results are probably accurate to one or two percent of the given values. The $K\alpha$ lines from a molybdenum water-cooled tube served as a source of x-radiation.

The thicknesses of the films were calculated from the time of sputtering, from the results obtained by weighing the film deposited upon another piece of glass sputtered for 30 minutes as a basis for the calculations. The thickness of this film was found to be 1.23×10^{-5} cm. The results of the measurements are tabulated as follows:

TABLE I. *Variations of critical angle with thickness of nickel films.*

Mirror No.	Time of sputtering (min.)	Critical angle (radians)	Color of film	Thickness (cm $\times 10^6$)
1	0	0.0040	white	0.0
2	2	0.0043	white	0.08
3	3	0.0043	light yellow	0.12
4	4	0.0043	yellow orange	0.16
5	5	0.0041	purple	0.205
6	6	0.0039	blue	0.25
7	10	0.0037	white	0.41
8	50	0.0034	white	2.05
	Value calculated from dispersion theory	0.0034		—

In order to show the manner in which the critical angle varies with the thickness of the nickel film (expressed in terms of sputtering time) the above results have been plotted together with the previous results obtained by sputtering the nickel films directly upon glass without the intervening film of platinum.

² Edwards, Phys. Rev. **30**, 91 (1927).

An inspection of the plotted values of the critical angle shows that the proper thickness of the film necessary to give the correct value of the critical angle is approached logarithmically. It is to be inferred from the character of the two curves that any theory which might be proposed to explain this phenomenon would probably indicate that an infinite thickness of the reflecting material would be necessary in order to obtain a correct value of the critical angle. Within the ordinary limits of error in measurement, this limiting value of the necessary thickness would, as the evidence presented shows, be reached very much sooner.

The above experimental results permit of a rather simple theoretical explanation. If we assume that the path of the x-radiation in the reflecting substance is along the line of the incident beam, since the index of refraction is very nearly equal to one, and that the scattered radiation is in line with the reflected ray, then we may determine the total length of the path in the metal

traversed by the radiation from any underlying electrons. The intensity of the radiation from any scattering electron, as it emerges from the reflecting surface, will depend upon the intensity of the incident beam, the absorption coefficient of the reflecting material and the length of the path traversed in the metal. If an electron is to contribute any measurable intensity to the reflected ray and therefore participate in the phenomenon of reflection, it must not lie too deeply in the metal.

If the distance of a contributing electron below the reflecting surface is indicated by the symbol d and the critical angle (in radians) is called c , then the length, x , of the path in the reflecting medium which the radiation must traverse is given by $x = 2d/c$. The intensity of the emerging radiation will be expressed by the relation

$$I = I_0 e^{-\mu_m \rho x}$$

where the symbols have their usual meaning. With these two equations we may therefore determine the necessary thickness of a film in order to have the contribution by the deepest electrons too small to affect the intensity of the reflected ray. If the critical angle may be made with a measurement that has an error of 1 percent, then the intensity of the radiation from the deepest electrons should be less than 1 percent of the intensity of the incident beam. We could therefore assign a value of 100 to the ratio I_0/I .

We may apply this explanation to the experimental data given above. The values of the measured critical angles are probably accurate to better than a 1 percent error. Putting $I_0/I = 100$ and taking the mass coefficient

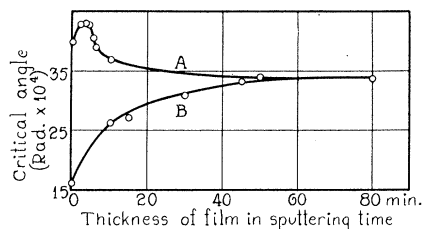


Fig. 1. Experimental curves showing the dependence of the critical angle of nickel films upon the thickness of the film and the underlying material. Curve A, nickel over platinum. Curve B, nickel over glass.

of absorption of nickel at 48.2 for the radiation used, and $\rho = 8.75$ gm/cc, the calculated value of x is found to be 0.011 cm. The correct value of the critical angle for nickel when using the $K\alpha$ radiation from molybdenum is probably 0.0034 radians. From these values d comes out to be equal to 1.87×10^{-5} cm, which is in close agreement with 2.05×10^{-5} cm, the thickness of the nickel film on platinum which gave a correct measured value of the critical angle.

Attention should be called to one other result in the measurements of the critical angle obtained from the four thinnest nickel films on platinum. Each of these gave a value which was greater than that of the bare platinum. These results would seem to indicate that the electronic density of the combined metals is greater than that of either the nickel or platinum alone. If this is the case one would expect that the first nickel atoms deposited upon the platinum are embedded among the platinum atoms producing an alloy having a greater density than the platinum alone. The fact that the nickel films adhere tenaciously to the platinum films supports this viewpoint.

UNIVERSITY OF CALIFORNIA AT LOS ANGELES,
January 15, 1929.