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OPTIMIZATION OF THE INTERFERENCE LAYER METALLOGRAPHY FOR AUTOMATIC IMAGE ANALYSIS AND ITS APPLICATION TO HIGH TEMPERATURE ALLOYS

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ABSTRACT

Conventional metallographic procedures usually yield sufficient contrast just to distinguish precipitates from the metallic matrix. However, to discriminate for example between different types of carbides these procedures do not always supply satisfactory results - especially if microstructural analyses by automatic analyzers are required. To increase the contrast, interference layers are deposited on the polished section. In order to optimize this procedure criteria for the selection of suitable interference layer materials and specifications for the layer-thickness and wavelength of the direct illumination have been determined.

INTRODUCTION

The generation of a sufficient contrast plays a key role in specimen preparation for the characterization of multiphase metallic and non-metallic materials. In this connection interference layer metallography (Pepperhoff and Ettwig, 1970), i.e. intensification of the contrast by applying thin, absorption-free or absorbing layers to the metallographic section of the material sample, has proved to be an extremely efficient procedure. The optical system can as a rule be adjusted in such a way that sufficient contrast values result between all the phases involved. The purpose of interference layer metallography is

- to make the individual structural components distinguishable to the human eye and to identify them on the basis of their luminosity, colour and colour saturation;
- to make the individual phases of a material accessible to quantitative structural analysis by the application of monochromatic light.

METHOD

The deposition of interference layers on polished metallographic sections makes it possible to intensify the contrast between different phases considerably. Maximum contrast (K=1, where K is the relative difference in reflectivity) is present if the reflectivity of one structural component is completely deleted (Bühler and Hougardy, 1979). This condition can be used as a criterion for selecting a coating substance. Theoretically, not only one single but any number of coating materials are suitable for reducing the reflective power of a given phase with optical constants n_p = refractive index and k_p = absorption coefficient. This can be clearly seen from Fig. 1. Curves for completely extinguished reflectivity (R = 0) for 5 different coating materials (characterized by the optical constants of the coating n_z and k_z) are plotted here as a function of the optical constants of the phase n_p and k_p. The determination of optical properties has been published elsewhere (Schmidt et al., 1982). The curves are computed according to the formula given by Heavens (1965) in such a way that they intersect at the point $n_p = 1.5$, $k_p = 2.5$ - thus the 5 coating materials upon which the diagram is based are suitable as interference layers for a phase with these optical constants.

If the optical constants of these 5 coating materials qualified for a given phase are plotted in an n_z-k_z diagram (▲) then surprisingly enough an almost linear coherence results (Fig. 2), which also permits an interpolation between these values. If at the same time the measured values for the layer material (e.g. iron oxide) are plotted in a diagram of this type (●) the best suitable coating conditions can be directly specified. In the example given here the phase $n_p = 1.5$ and $k_p = 2.5$, which is representative of a certain carbide in a nickel base alloy iron layers deposited by sputtering would be suited best at a wavelength λ of 565 nm. The coating thickness d_z (◆) required can also be deduced from this diagram. It increases with rising absorption coefficient k_z of the layer.

Diagrams of the type shown here are determined for a relatively wide spectrum of n_p-k_p values and for different layer materials so that the maximum contrasting coating, the wavelength λ of light and the layer thickness could be immediately derived for any phase. Thus, the interference layer procedure is optimized not only for iron and nickel base alloys but also for any metallic or ceramic material.

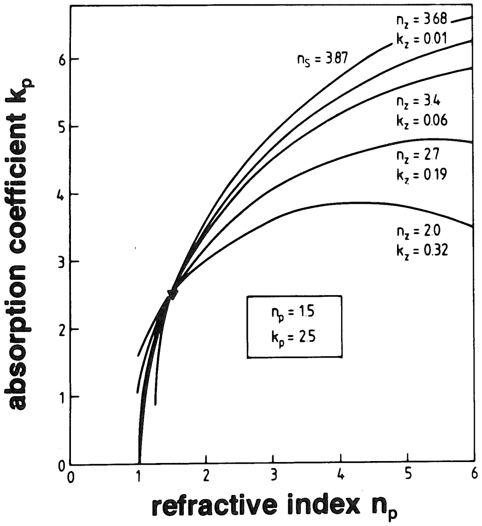


Fig. 1: Curves of zero reflectivity in dependence on absorption coefficient k_p and refractive index n_p of the phase for 5 different layer materials. The reflectivity of a phase with optical properties $n_p = 1.5$ and $k_p = 2.5$ can be deleted with all these layers, hence yielding maximum contrast conditions with other phases.

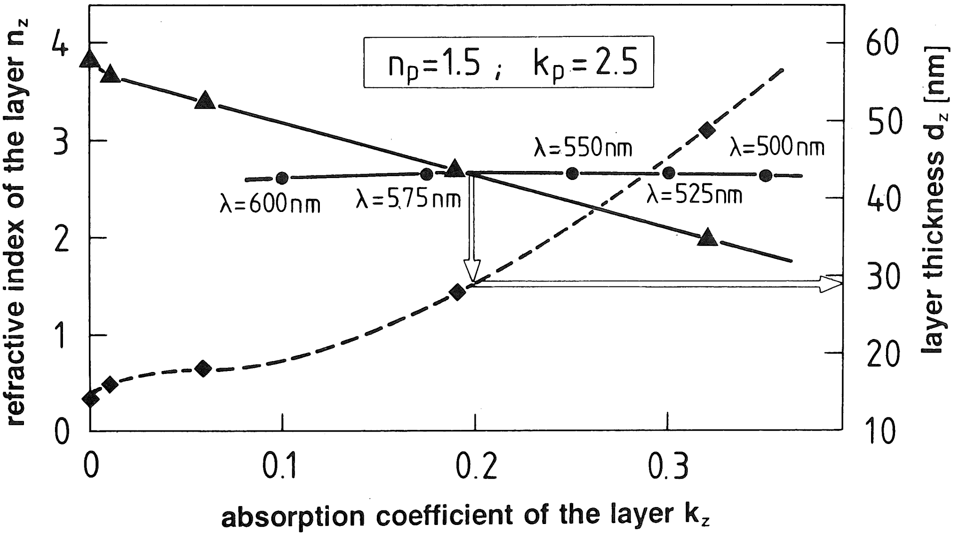


Fig. 2: The straight line (▲) represents all possible n_z- and k_z-values that make it possible to reduce the reflectivity of a phase $n_p = 1.5$, $k_p = 2.5$ to zero. The almost horizontal line (●) shows the optical properties of an iron oxide layer (determined on a glass substrate) at different wavelengths. The point of interaction characterizes the best suited coating conditions; i.e. a wavelength of 565 nm. The broken line (◆) represents the required layer thickness.

DISCUSSION

To demonstrate the reliability of this procedure under realistic conditions a Ni-base-alloy, HASTELLOY X, has been coated with an iron-oxide layer by sputtering. In Fig. 3 curves of minimum reflectivity for an iron-oxide coating have been calculated (similar to Fig. 1) as a function of the optical constants of the phase n_p and k_p. The chain-dotted line represents the zero-reflectivity curve (R = 0 %). The left diagram has been calculated for a wavelength λ = 500 nm with optical constants of the coating n_z = 2.61, k_z = 0.35; in the right diagram the wavelength has been increased to λ = 600 nm thus representing different optical properties of the coating (n_z = 2.60, k_z = 0.1). In addition the measured n_p,k_p-values for the phases of the alloy (matrix, carbides of the type M_6C and $M_{23}C_6$) are indicated.

Since the absorption coefficient of iron oxide depends strongly on the wavelength λ of the light (see Fig. 2), the zero reflectivity curve in Fig. 3 becomes less steep with increasing λ . At λ = 500 nm the k_p,n_p-values of the single phases of HASTELLOY X lie below the zero-reflectivity curve; at λ = 600 nm the position is reversed. Hence in between the wavelength limits λ = 500 nm and λ = 600 nm one should expect 3 specified wavelengths with define the conditions for R(matrix) = 0, R(M_6C) = 0 and R($M_{23}C_6$) = 0. This fact is confirmed in Fig. 4 presenting a metallographic section of HASTELLOY X coated with an iron-oxide interference layer. The three micrographs show the same position at 3 different wavelengths indicating zero-reflectivity for the single phases: at λ = 500 nm the matrix appears black, at λ = 520 nm the reflectivity of the $M_{23}C_6$-carbide has reached a minimum and for λ = 560 nm the carbides of the type M_6C are extinguished.

The criteria for selecting the optimum interference layer presented in this paper aimed at achieving maximum contrast ratios. In practice however the contrast, K = 1, cannot be achieved because the individual phases in general are not extinguished completely (see Fig. 4). Significant deviations in the predicted contrast and in the predicted wavelengths results

- because no light source with strictly monochromatic light is available for microscopy,
- because the interference layer cannot be applied with precisely the precalculated layer thickness,
- because the optical constants required for optimizing the system cannot be measured without error,
- because all the parameters involved in the system (layer thickness, optical constants) are subject to relatively large fluctuations,

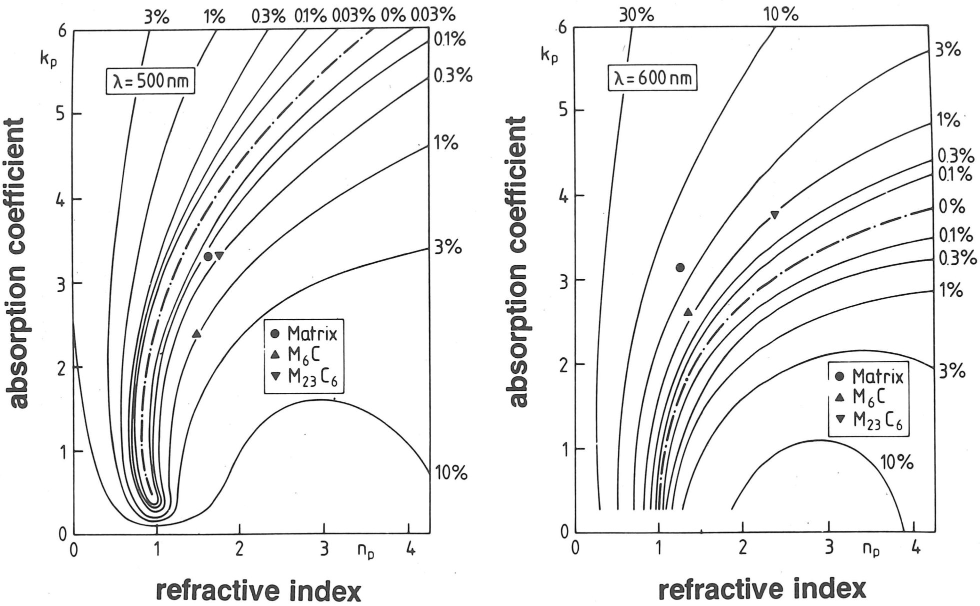
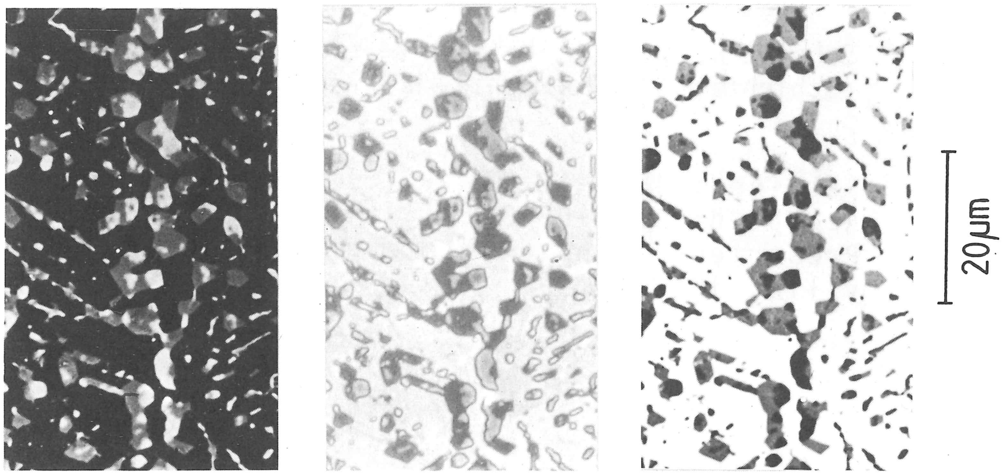


Fig. 3: Curves of minimum reflectivity for an iron-oxide coated metallographic section in dependence on absorption coefficient k_p and refractive index n_p for two different wavelengths (λ = 500 nm left, λ = 600 nm right). The circle indicates the optical constants of the HASTELLOY X matrix, the triangles display the n_p,k_p-values of its carbides.



λ=500nm λ=520nm λ=560nm

Fig. 4: Metallographic section of HASTELLOY X coated with an iron oxide interference layer in monochromatic light.

- because variations in the chemical composition - which influence the optical constants - have a particularly serious effect.

Image analyzers working on the basis of TV cameras are able to resolve minimum contrasts in the range of K = 0.1 - 0.2 without any difficulty. It must therefore be noted that maximum contrast as a consequence of the error sources mentioned above must not fall below this limit. Therefore in practical work contrast values between the different phases of a high temperature alloy should be in the range of $0.2 \leq K \leq 1.0$. The interference layer procedure has proved to be very efficient even under extreme conditions - i.e. if two phases with almost equal optical constants are to be separated.

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