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Application of Differential Interferometry with Two Polarized Beams

The purpose of this Letter is to show how the technique of differential interferometry can be used to decided advantage for examining metallurgical specimens in cases where ordinary bright-field and phase-contrast techniques are inadequate.

The metallographic microscope is a primary tool in every field of material development, from solid-state physics (semiconductors and thin films) to metallurgy. The conventional metallographic microscope, however, proves inadequate in such cases as (1) to separate very close details; (2) to single out details offering very slight contrast with the matrix; and (3) to single out minute differences in level.

The electron microscope, of course, can be used to overcome these difficulties, but the preparation of samples is very elaborate, the areas that can be examined are most limited and, moreover, many laboratories are not equipped with such instruments. For these reasons a number of devices adaptable to metallographic microscopes have been developed to extend their potentialities in the directions of higher contrast and relief.

Two methods are available to enhance contrast: phase contrast and interferential contrast, the latter also having the capability of showing small differences in surface level.

As far back as 1935, Dutch physicist Zernike had developed a phase-contrast method. Since then, many devices based upon this method have been satisfactorily used. However, the phase contrast method requires that the observed detail be small—otherwise the light diffracted mixes too much with the direct light.

The interferential contrast principle was described by Sagnac in 1911. Owing to its very principle, this method does not discriminate between large and narrow details; moreover, the complexity of the devices had precluded the use of this method. Recently Nomarski[1] developed at the C.N.R.S. (Centre National de la Recherche Scientifique) a very practical method and related devices. This method allows better quality contrast and relief set-off than the devices previously used; moreover, it permits rapid measurement of level differences by interferometry.

We have equipped our Reichert microscope with these devices.

The object of this Letter is to show, by means of examples taken from various studies carried out at the Laboratory, the advantages this method offers in the study of structures and surface conditions.

First of all, it is necessary to indicate briefly the principle of the Nomarski method and device.

Principle of the differential interferometer

The Nomarski devices are based on differential interferometry. In the conventional interferometer, comparison is made between a wave, bearing the impression of the object under study, and a flat reference wave. The normal profile of the wave reflected by the object is thus determined. This device can be easily converted into a differential interferometer. For instance, if in the well-known Michelson interferometer, the surface to be investigated is put in the place of the input slit, and if the latter is lighted by a distant source, it can be shown that two waves are obtained in the image space which interfere as in the case of the conventional interferometer; but, while the latter gives the exact profile of the surface under study, the differential interferometer shows a differential profile. Thus, assuming profile A is the object under study (Fig. 1), the differential interferometer gives image profile B. The interference phenomenon observed is the same as produced in a conventional interferometer by an object profile similar to B, since in a conventional interferometer, the image and object profiles are similar.

Nomarski differential interferometer with two polarized beams

This device is not the optimum solution because the adjustment of the apparatus must be very accurate. Since the problem is to obtain two coherent beams with a low angular separation, it has proved of interest to use polarized light.

In order to be able to use a wide source, Nomarski was led to use a compensating prism, which allowed the device to be made more compact. A diagram of this interferometer is shown Fig. 1.

It is a *self-compensating interferometer* which offers the following advantages: high luminosity; ease of adjustment, even in the case of widely separated

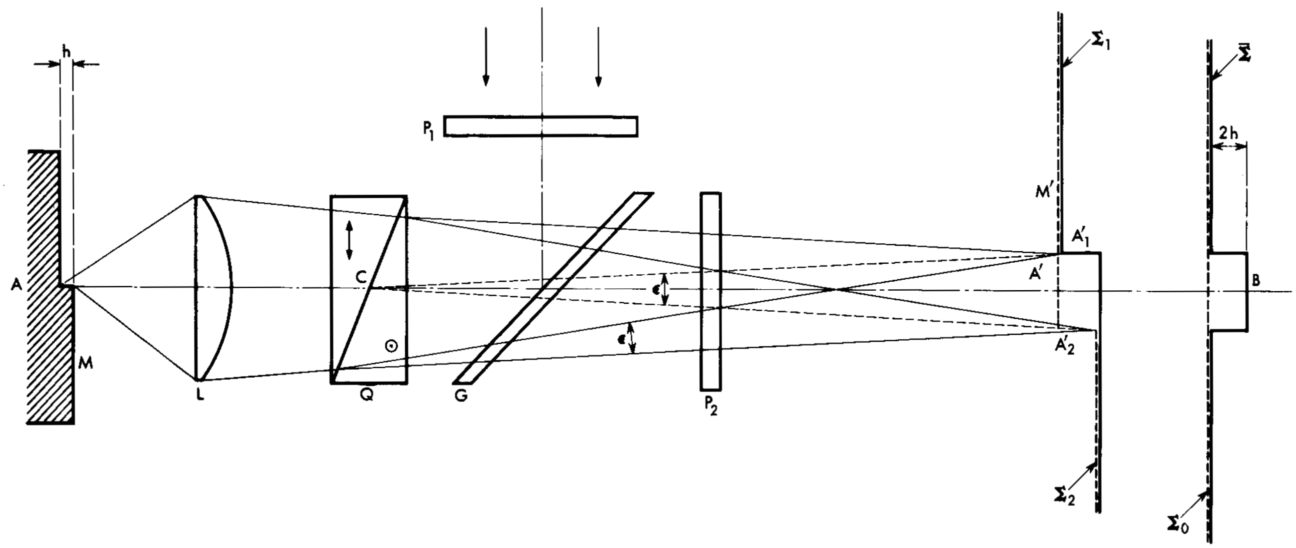


Figure 1 **The Nomarski Differential Interferometer.**

beams; very easy adaptation to a metallographic microscope equipped with polarizers.

This device makes it possible to measure level differences either using fringes of equal thickness, or uniform tint interferences (by inserting a compensator in the diagram).

We use the first method, in which fringes parallel to the prism axis appear in the image field if the prism is not located exactly in the focal plane of lens L. The number of fringes visible in the field is proportional to the distance from the prism to the focal plane of the lens, and to angle α of the prism. It is thus possible to vary the interfringe distance, by varying the distance from the prism to the focal plane, for a prism having a given angle.

Application to the measurement of thin-film thickness

Sample preparation is the same as for thickness measurement by multiple-beam interferometry: the deposit, the thickness of which is to be measured, is made only on one half of the substrate. Then, a layer of a highly reflecting metal is deposited on the whole surface of the substrate.

The sample is then placed on the stage of a metallographic microscope, and, after the Nomarski device has been inserted between the objective lens and the illuminating mirror, the sample is examined in polarized light.

For a perfectly plane surface, straight fringes appear in the eyepiece, and the spacing between these fringes may be varied by increasing or decreasing the distance between the prism axis and the focal plane of the lens by means of a lever. Sharper fringes are obtained with a small magnification and we generally use the 8× lens.

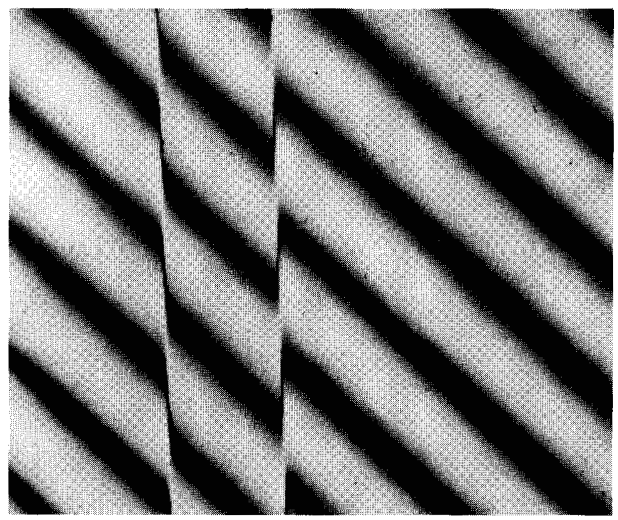
When there is a difference in levels, a fringe offset occurs, as shown on Fig. 2. More precisely, a double offsetting occurs, as explained above (profile A of Fig. 1 converted into profile B). Measuring offsetting d, and interfringe distance i, makes it possible to determine thickness e of the thin film:

$$e = \frac{\lambda}{2} \frac{d}{i} .$$

In order to obtain a sharp offsetting, it is necessary that the thickness of the second deposit be low. That is why aluminum is used preferably to silver, although its reflecting power is slightly lower. In fact, Al permits opaque layers to be obtained for thicknesses between 300 and 400 A, instead of 900 to 1000 A with Ag.

For our measurements, we use as a source of monochromatic light a sodium vapor lamp ($\lambda = 0.589\ \mu$)

Figure 2 **Measurement of thin film thickness.**



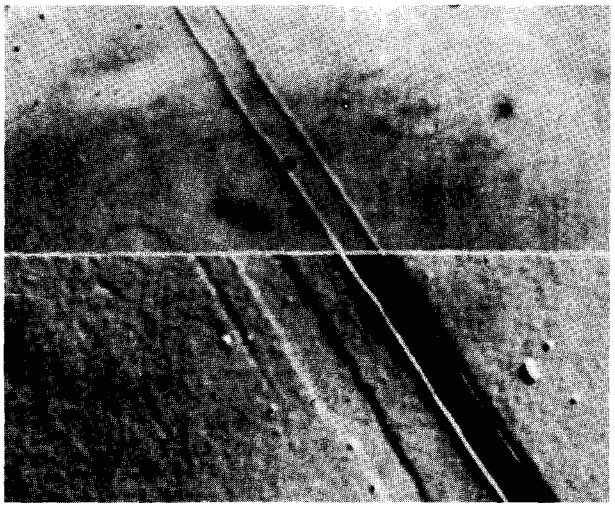


Figure 3 **Thin film surface defects (1000×).**

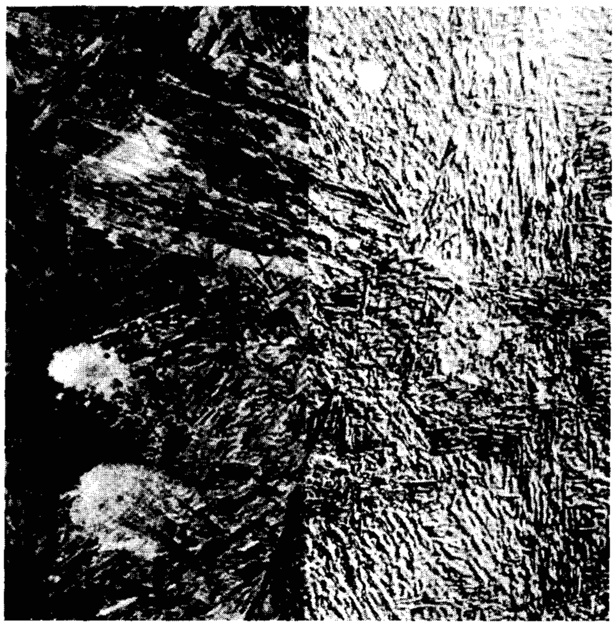
which gives high-contrast interference fringes. The measurement of i and d is effected during observation, using an eyepiece whose parallel reticular lines can be moved with respect to each other by means of a graduated micrometer screw.

This method makes it possible to carry out very accurate measurements ($\pm 1.5\%$ for thicknesses of the order of 2000 A).

Two variations of the technique should be noted: (1) When the deposits are thick, it may be very difficult to follow the offsetting of a fringe in monochromatic light. It is then possible to measure the offsetting of the center black fringe in white light and the interfringe distance in monochromatic light. (2) By rotating the analyzer by 45°, the sample can be observed in a bright field, which gives the method an additional advantage.

It should be noted that the photograph in Fig. 2, for reasons of light intensity, was not taken in sodium vapor light, but in white light in conjunction with a yellow filter whose bandpass is wider, hence the lack of sharpness of the fringes.

Figure 4 **Hardened Cr-Ni-Mo steel (42 CD 4) (500×).**



Interferential contrast device

The interferential contrast device is derived from this differential interferometer—the problem was to reduce the distance between the twin images given by the lens and the prism. Twinning must be sufficient so that diffraction cannot affect the contrast of the edge of the detail to be observed, but must be low enough to avoid blurring the detail contour. For this purpose it is sufficient that the distance between the two images be equal to twice Lord Rayleigh's resolving limit. A very simple calculation shows that this condition is met in the usual objective lenses, if

$$38' < \alpha < 76' \qquad (\alpha\text{: prism angle})\,.$$

But it is necessary for the self-compensating device that the pupillary fringes be localized in the focal plane of the lens. That is why, to be able to use medium and powerful objective lenses, the prism of Fig. 1 is replaced with a double prism composed of a prism whose axis is parallel with the edge, and a prism whose axis is highly slanted with respect to its sides, while remaining perpendicular to its edge.

The advantages of this device are the same as those mentioned for the interferometer, including the possibility of switching from interferential-contrast observation to bright-field observation, without modifying the focus adjustment, merely by rotating the analyzer 45°.

Applications

The use of this device was most profitable whenever bright field and phase-contrast observation proved inadequate. In fact, a comparison between the phase-contrast method we had been using for several years and the interferential contrast method with two polarized beams, shows that the latter not only obtains much better results (the structure, relief and contrast are set off much more sharply[2,3]) but also can be used much more simply and rapidly.

The fields in which the Nomarski device proved most efficient for solving our problems are the following:

• 1. *Examination of surface conditions*

This process is particularly adequate for the examination and evaluation of the quality of highly polished surfaces. In this case, bright-field observation cannot sense low-amplitude defects, and does not permit

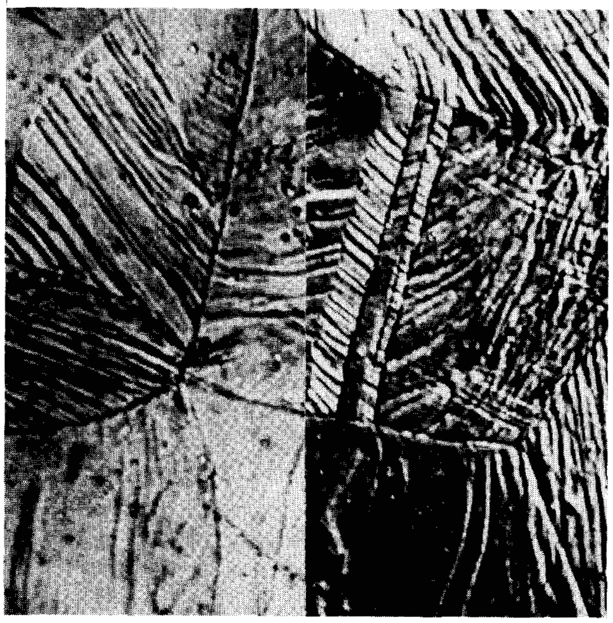


Figure 5 **Cold-worked bronze (1000×).**

comparison, for instance, of two surfaces prepared under slightly different conditions. Interferential contrast with two polarized beams shows very sharply any difference in level, however small it may be.

This method is particularly valuable for the development of electropolishing and electroplating processes, and for the examination of thin films deposited on glass supports.

Figure 6 **Micrographic section of a transistor (500×).**

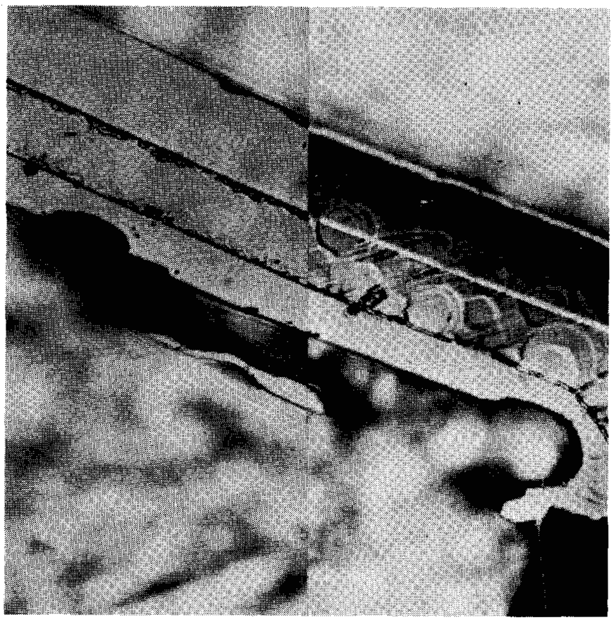


Figure 3 shows a micrograph of a sample prepared for the measurement of the thickness of deposits obtained by vacuum evaporation.[4] A number of defects have been produced in this sample, some of which can be seen only by interferential-contrast observation. On the right of the double line, the over-all thickness is about 1600 A. The double line is due to a slight displacement of the mask during the first deposit. Therefore, there is an intermediate thickness between the two lines. In bright-field observation, these lines are practically the only details that can be seen. By the interferential-contrast method, on the contrary, one sees very clearly not only the asperities due to dust and a defect in the substrate, but also the surface finish of the glass plate.

It is often very desirable to complete the examination of surfaces in interferential contrast with the observation of interference fringes.

2. *Examination of hardened structure (obtained by quenching or by work hardening)*

When examining hardened steel, the structure appearing on bright-field after etching is often lacking in sharpness. While in most cases the direction of the needles can be determined, it is very difficult to infer accurate information about their length and shape. These details are needed in order to evaluate the quality of the treatment and to distinguish between the different acicular structures, from martensite to upper bainite. With the interferential contrast method, on the contrary, all details appear very sharply (Fig. 4: Ni-Cr-Mo steel), and it is possible to discriminate between the various structures and to check whether the material does not contain undesirable phases: ferrite, austenite, et cetera.

In the case of cold-worked structures, interferential-contrast observation makes it possible to distinguish very sharply the slip-line network. Figure 5, a micrograph of a cold-worked bronze, is a good example.

3. *Structures obtained by diffusion processes*

It is often essential to check the quality of a diffusion operation by observing structure changes (phase change, or creation of new phases, recrystallization, et cetera) or any associated defects, such as porosities due to different diffusion speeds of the two elements, formation of detrimental compounds at grain boundaries.

Figure 6 shows a section of a transistor. While in the bright-field micrograph, recrystallization layers are clearly seen, interferential contrast shows a great many other details. In particular, the latter method makes it possible to distinguish in a recrystallized layer, parallel with the recrystallization front, a line whose presence is easily explained by the diffusion process, but which does not appear in the bright-field micrograph.

These few examples show the great advantages derived from the use of these devices in our laboratories.

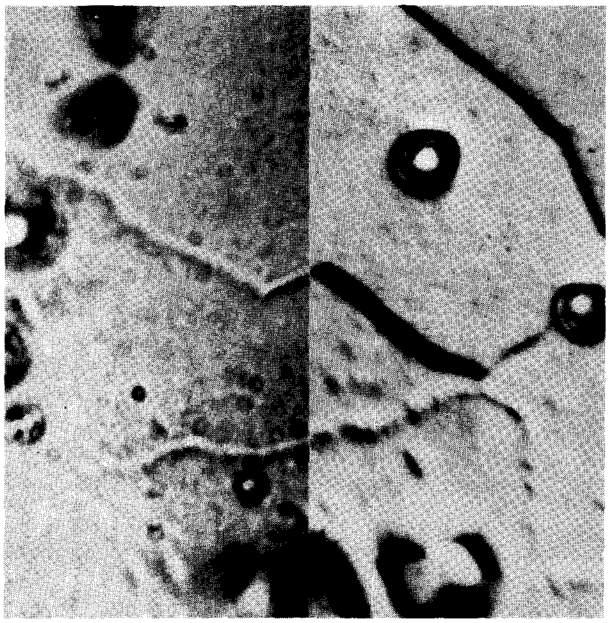


Figure 7 **Sintered iron (1500×).**

In other fields, their interest may be at least as great. An example is the examination of dislocations. Thus, in an interferential-contrast micrograph of a sintered iron sample, (Fig. 7) the present grain boundaries, which appeared after annealing, are clearly differentiated from the former ones, which appear as dotted lines; in the bright-field micrograph, the difference is practically not visible.

This possibility of observing dislocations and corrosion patterns seems of particular interest, as evidenced by recent research.[5,6]

References

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4. In this micrograph, and the following, there is a continuity between the area taken on bright field and the area in interferential contrast.
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