An application of differential interference contrast in metallographic examination

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Abstract: As one of the most exciting inspection and powerful analysis methods in modern materials metallographic examinations, the difference interference contrast (DIC) method has many advantages, including relatively low requirement for specimen preparation, obvious relief senses observed under microscope. Details such as fine structures or defects that are not or barely visible in incident-light bright field, could be easily revealed and thus make materials analysis more reliable. Differential interference contrast produces an image that can be readily manipulated using digital and video imaging techniques to further enhance contrast. But, studies of material metallography based on DIC method have rarely carried out. Based on the fundamental principle of the DIC method combing with the computer image analysis, applications of DIC method in materials metallographic examination were investigated in this study.

Keywords: difference interference contrast method; metallographic examination; and image analysis

CLC number: TG14, TG115.2

1. Introduction

The difference interference contrast (DIC) method is one of the most exciting inspection and powerful analysis methods in modern materials metallographic examinations [1], which has many advantages. ① Specimens preparation is relatively simple. For certain specimens, their microstructure could be observed without etching under the microscope by using the DIC method as the polished specimen surface is preserved. ② The observation of the specimen surface via the microscope has obvious accidented senses, taking the form of relief. The relative locations of different features in a specimen could be distinguished readily. The particles, crackles, caves, slopes, valleys, and other discontinuities could be judged correctly, with the improved accuracy of the metallographic examination, in which the contrast of the features are enhanced. ③ With the DIC method, details such as some fine structures or defects that are not or barely visible in incident-light bright field could be easily observed. ④ The DIC method based on the traditional polarized light with a polarizer and specialized beam splitting prisms, named Wollaston or Nomarski prisms, adds pseudo-color which improves visual contrast between different phases. In addition, differential interference contrast produces an image that can be easily manipulated using digital and video imaging techniques to further enhance contrast.

Like polarized light, DIC was also primarily developed as an analytical method to determine various optical properties of crystals. Thus, it is a method of measurement, requiring specific knowledge and specially-designed microscopes of considerable complexity. Appropriate applications of the DIC method can yield fine details of microstructure.

Differential interference contrast has found wide application in biology due to its simplicity in use and emergency of commercially-available microscopes. However, little study of materials metallography based on the DIC method has been carried out. This is because most laboratories for materials research presently are not equipped with metallographic microscopes including DIC optical components. Few materials researchers have realized the potential and capability of the DIC method microstructure analysis.

The basic principles of the DIC method are introduced based on the Neophot32 microscope in this paper. Combining with the technology of computer image analysis, applications of the DIC method in materials metallographic examination are presented.

2. The fundamental principles of DIC Method

The DIC method is based on the theory of differential interference contrast (DIC) by using a Nomarski prism in the polarized light fields. The optical path is very sensitive to small height difference of light interference in nanometer size.

The basic DIC system, first devised by Francis Smith in
1955, is a modified polarized light microscope with two additional Wollaston prisms added, one at the front focal plane of the condenser and the second at the rear focal plane of the objective. Several years later, Georges Nomarski, modified the standard Wollaston prism configuration to enable these exceedingly thin optical components to be physically located away from the conjugate planes of an aperture.

The optical components required for differential interference contrast microscopy do not mask or otherwise obstruct the objective and condenser apertures, thus enabling the instrument to be employed at full numerical aperture. The result is a dramatic improvement in resolution (particularly along the optical axis), elimination of halo artifact. Moreover, differential interference contrast can produce images that can be easily manipulated using digital and video imaging techniques to further treatments or quantitative analysis.

2.1 The basic components of DIC method

In general, only four basic components are required to configure a research or standard laboratory bright field microscope for observation in differential interference contrast: linear polarizer, sensitive wave plate (also termed λ-a compensator), DIK module and analyzer.

Linear polarizer is inserted into the optical pathway between the microscope light port (or anywhere after the illumination source collector lens) and the condenser lens assembly. This component is designed to produce the necessary plane-polarized light for interference imaging. The vibration plane transmission axis for the electric vector component is oriented in an East-West direction (right to left when standing in front of the microscope), typical of a standard polarized light microscope. Some differential interference contrast designs incorporate a rotating polarizer combined with a quarter-wavelength retardation plate at this position in the microscope.

DIK module is provided with a Nomarski prism which consists of two prisms at right angle slidable in its working position and two set-screws. By actuating the smaller set-screw towards the optical axis of the microscope, an optimum position of the prism relative to the objective's exit pupil; while actuating the larger set-screw, the relative position of the two prisms of the Nomarski prism resulting a slight change of the optical path difference that producing the interference effect could be adjusted and the contrast could be varied delicately. The Nomarski prism separates the polarized light emanating from the polarizer into it. Incident wavefronts of plane-polarized light are split (or sheared) into mutually perpendicular (orthogonal) polarized components (termed ordinary and extraordinary wavefronts) by the Nomarski prism, and then pass through the specimen. The material of the prism is normally quartz.

Analyzer, the second linear polarizer, is installed behind the Nomarski prism, usually in an intermediate tube between the microscope nosepiece and observation (eyepiece) tubes. Termed an analyzer, this polarizing element is positioned in the optical pathway before the tube lens (for infinity-corrected microscopes) and image plane. The analyzer is oriented with the transmission axis of the electric field vector perpendicular (North-South) to that of the substage polarizer. Components of circular and elliptically polarized light arriving from the objective prism pass through the analyzer and subsequently undergo interference to generate the DIC image at the microscope intermediate image plane.

Because only the second series have rich hue where the optical path difference is between 560 and 1 120 nm. Normally, a piece of full-wave plate should be added in the optical path of polarized microscope. This full-wave plate can produce an optical path difference of 576 nm. Regardless of the polarization state changes, it can extend the wide range of color contrast nuances gradually and always produce bright color changes. This full-wave plate is called the sensitive plate or λ-a compensator.

2.2 The imaging mechanisms of DIC method

Fig. 1 presents the basic Nomarski prism differential interference contrast device. When light passes through an optically homogeneous medium, it produces one refraction of light. But when it passes through a non-homogeneous material, the refracted light will be split into two bundles, which is called the birefringence, or double refraction. The vibration directions of the two lines produced by the birefringence are perpendicular to each other. Their speed is different and their refractive index is different.

When a bundle of parallel light passing through the polarizer, it will become plane-polarized light. Then the light after polarizing passes through the DIC prism, it splits into two lights. One, being called ordinary light or o light, propagates along its original direction. The other, being called extraordinary light or e light, deviates slightly from its original direction. Both the o and e lights produced by the birefringence are polarized lights.
Nomarski prism, normally using quartz, is a revised Wollaston prism. It consists of two prisms at right angle. Cutting the crystal along certain lattice plane and then grinding, the two parts were glued by turning one of them 90° with respect to the other. Different from Wollaston prism, the optical axis of the first prism of Nomarski is parallel with the cutting plane. The refractive index of ordinary light \( n_0 = 1.544 \) and the extraordinary refractive index \( n_e = 1.553 \) are stable for any light with different wavelength. Because the difference between \( n_e \) and \( n_0 \) are very small, the angle of two plane-polarized lights produced by the prisms is very small.

In Fig.2, when natural light illuminates the AB surface vertically, the ordinary light would coincide with the optical axis but the extraordinary light is bent with an angle \( \alpha_1 \). They propagate with different velocities \( v_o \) and \( v_e \). Because the optical axis of second prism is perpendicular to the first one, when they enter the second prism the ordinary light becomes extra-ordinary light (or oe light) and the extraordinary light becomes ordinary light (or eo light). After passing through the second prism, the two lights become two plane-polarized lights with very small angle and their direction of vibration is perpendicular each other.

Since the velocity of \( o \) light is larger than that of \( e \) light, the two plane-polarized lights should produce different optical path difference after going through the wave plate. In other words, in this light path system, the optical path difference \( T_1 \) (about several nanometers) is the distance that the faster light passes over the slower light.

The analyzer admits only the components of the two lights that are parallel to the analyzer. When the two differential lights pass through the objective lens to reach the uneven surface parallel, they are reflected by the surface and then pass through the objective lens again. But the location where the lights reenter the Nomarski prism is different. The two differential lights should have some optical path difference \( T_2 \) due to the uneven surface, crack, micropore or grain boundary and the changes of light wave phase resulting from the different refractive index of different phases. When the two lights reenter and pass through the Nomarski prism again, an additional optical path difference \( T_3 \) is produced. The two lights are still two plane-polarized lights of which direction of vibration is perpendicular each other. Before entering into the analyzer, the total optical path difference \( T_{total} = T_1 \pm T_2 \pm T_0 \).

Since the optical path difference can be expressed by integer times wavelength, then \( \sin(k\pi r) = 0 \) (where \( k=0, 1, 2... \)). The interference that yields two plane-polarized lights is equal to zero. That is equivalent to no light passing through the wave plate. Only when the light path difference can be expressed by integer times half wavelength, or \( (2k+1)/2 \) times the wavelength (where \( k=0, 1, 2... \)), then \( \sin((2k+1)\pi r) = 1 \), the two plane-polarized lights can pass through the analyzer and then interfere with each other, and the resultant amplitude of the wavelength becomes maximum.

As the Nomarski prism is shifted laterally, wavefront pairs contributing to the background become increasingly retarded and out of phase with respect to one another. As a result, the degree of elliptical polarization is increased in wavefronts entering the analyzer, and the background intensity progressively transits from black to medium and
lighter shades of gray. So as actuating the larger set-screw of the DIK module, the relative displacement of the two prisms of the Nomarski prism is generated and a pseudo three-dimensional relief image (where regions of increasing optical path difference (sloping phase gradients) appear much brighter (or darker), and those exhibiting decreasing path length appear in reverse) could be observed from the ocular (seen in Fig.3). The interference effect is zeroth-order interference now (according to the order of interference color and content of brilliance, the interference color can be divided into four series), and the dark shadow is often termed zero-order gray.

Different interference color corresponds to different optical path difference for illuminating parallel or orthogonal polarized light [5]. The range of optical path difference is between 0 and 1 680 nm. According to the order of interference color and content of brilliance, the interference color can be divided into four series (Table 1).

![Fig.3 The relief morphology of high manganese steel (Mn13)](image)

<table>
<thead>
<tr>
<th>Interference color series</th>
<th>Optical path difference [nm]</th>
<th>Interference color order</th>
<th>Interference color not produced</th>
<th>Boundary colors</th>
</tr>
</thead>
<tbody>
<tr>
<td>First order (zeroth order) series</td>
<td>0–560</td>
<td>from dark to steel gray, blue gray, white, yellow-white, bright-yellow, orange-yellow, red and purple-red</td>
<td>green and blue</td>
<td>very clear</td>
</tr>
<tr>
<td>Second order series</td>
<td>560–1 120</td>
<td>from purple to dark blue, sky blue, and green, yellow-green, yellow-orange and purple-red</td>
<td>purple and blue</td>
<td>clear</td>
</tr>
<tr>
<td>Third order series</td>
<td>1 120–1 680</td>
<td>from purple to blue, green-blue, sea green, green-yellow and lilac</td>
<td>not so clear than the second order series</td>
<td>no obvious boundary</td>
</tr>
<tr>
<td>Fourth order series</td>
<td>&gt; 1 680</td>
<td>from purple-gray to cyan-gray, green-gray, nattier blue-green, filbert-green and senior white</td>
<td>orange-yellow, red, purple, dark blue, sky blue, green, yellow green, yellow, orange, purplish red, violet, blue, green blue, sea blue, green, yellow, blood red, lilac, gray blue, and senior white</td>
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</tr>
</tbody>
</table>

Because the sensitive plate or λ-a compensator is added before the analyzer in the optical path of the microscope, and can produce an optical path difference of 576 nm (about a wavelength), the interference color order could be extended from the zeroth-order series to the second order series. When the optical path difference is zero, the interference color is dark. If the optical path difference increases gradually, the interference color changes from dark to steel gray, blue gray, yellow white, bright yellow, orange-yellow, red, purple, dark blue, sky blue, green, yellow green, yellow, orange, purplish red, violet, blue, green blue, sea blue, green, yellow, blood red, lilac, gray blue, and senior white (Fig.4).

Because the optical path difference increases continuously, the above order of interference color cannot be changed. The transition between colors is gradually. There is no significant boundary line. The higher the order, the less distinct the boundaries.
3. Applications of DIC in metallographic examination

With the DIC method, certain features such as structure details or defects that are not or barely visible in incident-light bright field could be easy to be judged and thus make materials characterization reliable.

The development of modern material science and technology requires detailed depiction of metallographic microstructure and accurate statistical analysis to the microstructure characteristic parameters, such as morphology of phases, quantity, distribution, phase size, phase area, etc. But the quality of the digital pictures captured by the digital devices (such as CCD, digital camera, etc.) depends greatly on the preparation of the sample in traditional bright fields. Also different phases in microstructure are shown at very close grey levels in traditional bright fields, which could result in great errors of measurements in quantitative metallography. Equipped with the differential interference contrast method, researchers could observe specific details of specimens that is not apparent on observed images captured by other methods.

3.1 Observation of fine structures

Fig. 5 shows the microstructure of a WC embedded coating on a machine tools fabricated by laser cladding method. The fine structure of the WC particles, such as their growth steps and cavities, can be distinguished clearly by the DIC method, while the WC phases are presented in white and the cavities are in black dots apparently.

Fig. 6 shows the microstructure of Ni3Al produced by the method of laser controlled synthesis reaction (incompact powder system, the scanning power of the laser is about 900 W, the scanning speed of the laser is about 2 mm/s). Observed in the bright field, the black phase is α-Ni, and the white phase presented in the form of petals is the eutectic structure of Ni3Al and α-Ni. No additional detailed information about this microstructure is obtained. However, while observed in the DIC, the growth sequence of the eutectic structure is presented.
3.2 Quantitative metallography of microstructure

Fig. 7 is the microstructure morphology of the gray iron (large white phase is iron phosphide eutectic in bright field). When calculating the amount of iron phosphide eutectic in the gray iron, the ferrite in pearlite may be counted in as it is present in a similar color with the iron phosphide eutectic in bright field, and thus a systematic error can be introduced. But in DIC, the iron phosphide eutectic is separated by a distinctive color with the ferrite in pearlite. Hence, the quantitative analysis of the iron phosphide eutectic becomes more accurate.

Fig. 8 is the quantitative metallography of the amount of ferrite in hypoeutectic Fe-C alloy fabricated by powder metallurgy method. In bright field, the color of the ferrite in pearlite is so close to the color of the free ferrite that a bias error can introduced when calculating the amount of ferrite by image analyzer. But in DIC, the free ferrite is rendered in evident color that can be distinguished easily with the ferrite in pearlite.
3.3 Calculation of height difference of the specimen surfaces

Because the interference color is produced by the optical path difference after the light illuminates the surface and nothing other than by light source. The optical path difference can be determined by the chromatograph table. Therefore, it is possible to use the changes of interference color to measure the height difference. Using the DIC method, the height difference of the specimen surface at mesoscopic level, such as the surface roughness of the component parts after precision finishing, the surface relief of martensite and bainite transformation and plastic deformation of the materials, could be determined.

Suppose the phase height difference \( h \) of the specimen surface is a stair step surface, it can be concluded from the basic principle of DIC method that the optical path difference \( A \) of a stair step surface produced by Nomarski prism is twice the height of the step, that is \( \Delta = 2h \). The height difference determines the optical path difference and the interference color (a chromatograph Table \([6]\) is plotted according to the optical path difference) or vice versa. The hue change of interference color is clear. But, the hue change of interference color can be used to determine the height difference since the brightness and saturation cannot reach the level of the light source. The isoheight contour according to the hue change of interference color of the DIC image can be plotted and the relationship between the hue change of interference color and height difference can be derived. J. Z. PAN and D. B. ZHU \([5]\) used the DIC method to examine the mesoscopic detail of crack tip deformation field. Fig. 9 is the mesoscopic details of crack tip deformation field in DIC.

4. Conclusions

As one of the most exciting inspection and powerful analysis methods in modern materials metallographic examinations, the difference interference contrast (DIC) method has many advantages. Due to relatively simple requirement for specimen preparation, obvious relief senses observed under microscope, details such as fine structures or defects that are not or barely visible in incident-light bright field, could be easily to be judged and thus make materials analysis more reliable. Differential interference contrast produces an image that can be easily manipulated using digital and video imaging techniques to further enhance contrast. Combining with the differential interference contrast method, researchers could observe specific fine microstructure about specimen that is not apparent from observing images captured by conventional methods. The potential of the DIC method has been evidently demonstrated in modern metallographic examinations.

References