Comparison of ULOI and Polarized Light Microscopy of Materials

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INTRODUCTION
Light microscopes may be used in many different ways depending on the purpose in mind. In all cases, the mode of illumination is chosen to focus the attention to the characteristics sought. ‘Normal’ illumination, conical illumination, dark-ground illumination, annular illumination, or oblique illumination, may be used. We may also adopt the methods of polarized light, phase contrast, stop contrast, or interference. The purpose of this article is to show that the unidirectional laser oblique illumination technique, a new mode of illumination in optical microscopy, is different.

POLARIZED LIGHT
Metallic surfaces can be divided into two kinds: first, optically isotropic where the optical properties are the same in all directions, and second, optically anisotropic where the optical properties vary for different directions. If an optically isotropic surface is illuminated with plane-polarized light at normal incidence, the light is reflected unchanged and can be completely extinguished by rotating the analyser. If, however, the light is not at normal incidence, the reflected light is elliptically polarized. Thus, it is not possible to obtain complete extinction with the analyzer in the crossed position. If the surface is optically anisotropic, the plane-polarized light incident normally on such a surface can be resolved into two components polarized parallel and perpendicular to a certain crystallographic direction and the phases and intensities of these two components may be changed by different quantities. Then, plane-polarized light is converted into elliptically polarized light. If the specimen is rotated about an axis normal to the plane of the sample, the surface shows four minima of brightness with four maxima between them [1].

SUMMARIZING, polarized light is used to distinguish between optical anisotropic phases and optical isotropic phases [2]. Optical anisotropic, or noncubic phases, exhibit their characteristic colours with four intensity peaks each, when examined in white linear polarized light (crossed Polaroids) by rotating 360°. Cubic metals which are optically isotropic do not give any effect under polarized light. They can, however, be made to react (1) by a deep etching, or (2) by the deposition of some film on the specimen surface. Jones [3] has shown that if an isotropic metallic surface is heavily etched the surface behaves in a similar way to an anisotropic surface when examined under polarized light. It is well known that typical geometrical etch pits, with distinctive patterns according to the crystallographic orientation, are formed in most metals and alloys when deeply etched. These etch pits are responsible for the observed polarization effects. The observation of the maxima and minima with crossed and uncrossed Polaroids led to the conclusion that the production of these maxima and minima might be the consequence of two successive reflexions at the metal surface. The behaviour of polarized light would point out to the fact that the surfaces of some crystals are really furrowed. This regular orientation is caused by chemical etching. This assumption is supported by the fact that in general with obliquely incident light the crystals appear brightly illuminated at two positions (separated by 180°) in a complete revolution of the stage. Under parallel Polaroids two maxima are found, but they are not sharply defined. These maxima coincide with the maxima obtained by using oblique incidence.

Anodized surface films on cubic metals, such as aluminium, are usually examined under polarized light to reveal grain contrast. Perryman and Lack [4] conducted an experiment to determine if the polarization response was due to surface contour effects or to the presence of an anisotropic surface film. They studied electrolytically polished zinc and cadmium (both anisotropic), electrolytically polished and anodized aluminium (isotropic), and Monel (isotropic) after a deep etching. These samples were examined before and after vacuum deposition of a thin film (80nm) of silver (isotropic) on the surfaces. Before deposition, all four samples responded to polarized light. After deposition, zinc and cadmium did not respond to polarized light. However, both anodized aluminium and the etched Monel still responded to polarized light. Therefore, polarization effects on anodized aluminium and etched Monel must be due to surface roughness effects and not to the presence of an anisotropic surface film.

UNIDIRECTIONAL LASER OBLIQUE ILLUMINATION
The ULOI technique was developed in our laboratory during the last decade. The illumination system of the microscope was replaced by a He-Ne laser beam of low power with an angle of incidence between 5° and 15° relative to the surface of the sample. The intensity, I, of the laser light dispersed by the crystalline grains varies as a function of the rotation angle, ω, of the sample around an axis perpendicular to its surface. The maximum of the I(ω) curve corresponds to a position parallel to...
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ized and no Polaroid sheet was being
resolved power of the objective. That is to say,
whether it exists or not, a main direction of the
surface pattern when the surface is furrowed.
As the angle of incidence is approximately 10˚,
the objective gathers up the highest diffracted
orders of the light dispersed by the topogra-
phy of the sample. When the intensity of the
light picked up by the objective is maximum for a given \(w\), we can infer that a main direc-
tion of the surface pattern exists and it is
located, for that \(w\), perpendicular to the pro-
jection of the laser beam on the surface.

One of the main questions that arose after
the first results obtained with ULOI was to
know if the physical phenomenon responsible for
the intensity curves was diffraction or polari-
ization, as some researchers suggested.
Therefore, different experiments were carried
out to answer the previous question. This arti-
CLE will compare the I(\(w\)) curves, obtained with
ULOI and polarized light illuminations,
by analysing crystalline grains of a chemically
etched sample of Zn, and in addition show that
the final results are similar to those obtained by O. Jones in 1924.

EXPERIMENTAL PROCEDURE
Experiments were carried out using polycrys-
talline samples of zinc, a Reichert microscope,
a 30 mw He-Ne laser (Melles-Griot). \(\lambda = 632.8\)
nm and non-polarized, and a Philips SEM 500
scanning electron microscope. The \(w\) angles
were measured using the goniometer of the
Reichert microscope. The intensity of the dis-
persed light was measured in one of the ocu-
lar pieces using a photometer (TIL 78 photo-
transistor). An objective of 8x was used for
acquiring the I(\(w\)) curves. Polaroid-type filters
were used for the polarizer and analyzer, in a
‘parallel’ position. The metallographic proce-
dure employed was: (a) mechanical grinding
using silicon carbide paper down to 600 grit
(granulometry of 15mm), and (b) chemical
etching using the following solution: CrO3
200g, Na2SO4 10g, HNO3 85 ml, H2O 1L.

RESULTS
A sample of zinc was prepared as a metallo-
graphic specimen. It was examined first under
unpolarized light in a microscope and then
under polarized light. The various crystals of
the metal could then easily be differentiated from one another by considerable differences in
intensity and colours. It was noticed that the
lower the numerical aperture of the objective,
the greater the contrast. When the sample
was rotated through one revolution, each
crystal alternately lightened and darkened
twice times [9]. The contrast was caused by the
formation of elliptically polarized light
depending on the orientation of the crystal.
Thus, zinc reflects plane-polarized light as
elliptically polarized light.

We then used the ULOI technique. Some
grains responded in a very notorious way to the
laser illumination. This fact indicated, in
agreement with previous studies, that such
grains had corrugated surfaces, which were
produced by the chemical etching. It was quite
difficult to discern, by using polarized light,
between grains that had a surface with fur-
rows from those that had a flat surface like a
mirror (Fig 1a and b). Clearly all the grains
respond to polarized light, but not all to ULOI
illumination.

Next, the intensity curves of different crys-
talline grains, which had a furrowed surface,
were analyzed. All the grains presented one
maximum of intensity in a rotation of 180°
with ULOI (remember that the laser was non-
polarized and no Polaroid sheet was being
used). Instead, the results obtained with polar-
ized light showed one maxima of intensity in
a rotation of 180° or two maxima in 360°, just
as our earlier studies established [3]. Similar
conclusions to those obtained by Jones were
verified. Therefore, the intensity curves
obtained with ULOI were the result of a dif-
fraction phenomenon. The objective of the
microscope gathered up part of the diffraction

Figure 1:
(a) Zinc specimen observed with polar-
ized light (b) Same area as (a) but
observed with unidirectional laser
oblique illumination (ULOI).
HPW = 760µm

Figure 2:
Intensity and rotational angle, \(\Delta \phi\) curves for a
crystalline grain of zinc. Only one maximum per
curve is shown.
pattern produced by the roughness of the surface. The variation of intensity of the light dispersed by the crystalline surface, as a function of $\psi$, was due to the rotation of the diffraction pattern around the specular beam.

Another difference between both illuminations was the resolution of the maximum (Fig 2). If the resolution, $D_\psi$, is established as the range of $\psi$ values around maximum value which have intensity values less than the error in the Y-axis, ULOI has a better resolution than polarized light. If an error of 0.025 (arbitrary units) is considered in the ordinate, the peak of intensity obtained with ULOI can be resolved within $\pm 2^\circ$, while in the case of polarized light, the resolution diminishes ($\pm 17^\circ$). All these data demonstrate the similarity between ULOI and oblique illumination [10].

Crystalline grains, which did not have a corrugated surface, did not show intensity curves (ULOI) with maximum values or peaks. This was verified by observing the grain surfaces with a scanning electron microscope. In Fig 3 the corrugated surface of two crystalline grains A and B can be appreciated. A deformation twin C is seen in grain A. The grains present intensity curves (ULOI) with very well defined maxima.

Figures 4a and b show another area of the Zn sample using polarized light and ULOI, respectively. Figure 4b indicates that there are three grains with a certain pattern on its surface. Thus, the total polarization effect is due to the optical anisotropy plus the faceted surface, which is produced by the chemical etching and depends on the crystalline orientation. It is clear that the polarization of grain D (Fig 4a) is due only to its optical anisotropy.

For an anisotropic material such as Zn, polarized light does not differentiate grains with and without a corrugated or faceted surface. ULOI distinguishes both types of grains clearly. These results allow us to conclude that the ULOI technique is not based on a polarization phenomenon.

CONCLUSIONS

All the crystalline grains responded to polarized light since Zn is optically anisotropic, but some of them presented furrows on the surface, which also produced polarization effects. It was difficult to distinguish, by visual observation, between these two kinds of grains by using only polarized light as a source of illumination. The ULOI technique allowed us to detect those crystalline grains having a corrugated surface, as well as determining the angular direction of the furrow with a precision of $\pm 2^\circ$.

In summary, the present study confirms first the conclusions obtained by O. Jones [3] and second that the $I(\psi)$ curves obtained with ULOI are not the result of a polarization effect as was suggested by other researchers; they are produced by the diffraction of the laser beam when impacting on the surface of the sample.

REFERENCES