



Design and Fabrication of Diffractive Optical Elements

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Diffraction by opaque screens



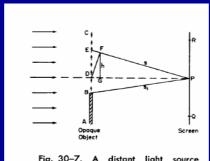
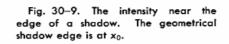
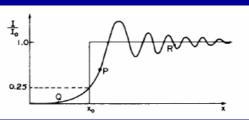


Fig. 30–7. A distant light source casts a shadow of an opaque object on a screen.



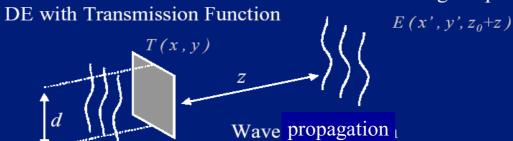






Function principle of DEs





Illumination Wave

 $E(x, y, z_0)$

The illumination wave is modulated by the diffractive Element.

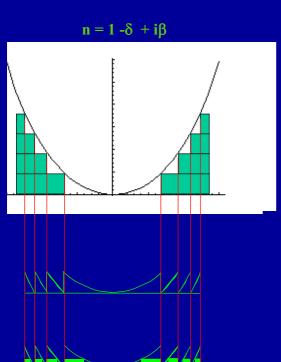
The modulated wave propergates through space and results in a certain desired signal wave.

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From Refractive to diffractive Optics





β: Imaginary part o complex refractive index:

λ: Incoming wavelength;

Refractive Profile

Kinoform Profile:

minimize absorption and simplify fabrication.

μ: Abs.
Coeffcient:

 $\Delta \phi$: Phase shift;

t: Material Thickness:

 $\mu = \frac{4\pi}{\lambda}\beta$

 $\Delta \phi = \delta(2\pi/\lambda)t = 2\pi$

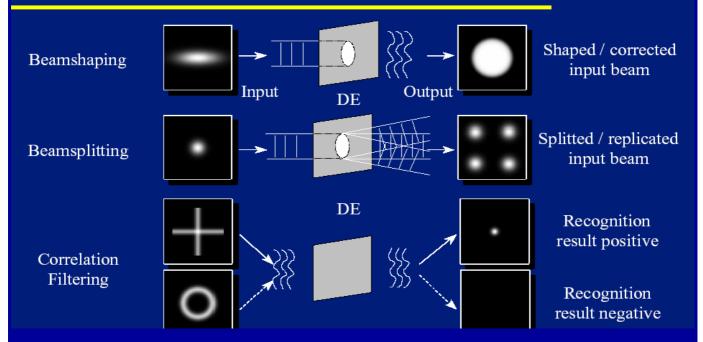
 $^{-1}$ H < ($\pi \Delta = \phi \Delta$)t

 $r \approx (f\lambda)^{1/2}$





Some Functions of Diffractive Elements



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Realisation of Microstructures



Index-modulated structure

Example: liquid crystal displays



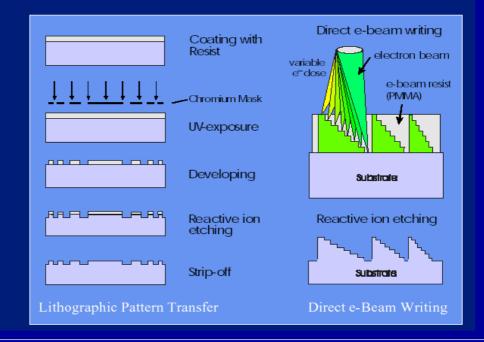
Surface-modulated structure

Example: fabrication by laser or e-beam lithography and etching





Fabrication Methods in Detail

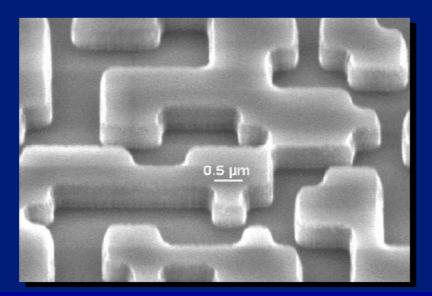


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Binary DE



Binary structure of a beamsplitter

Smallest structures: 0.5 µm!

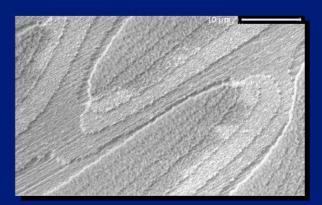




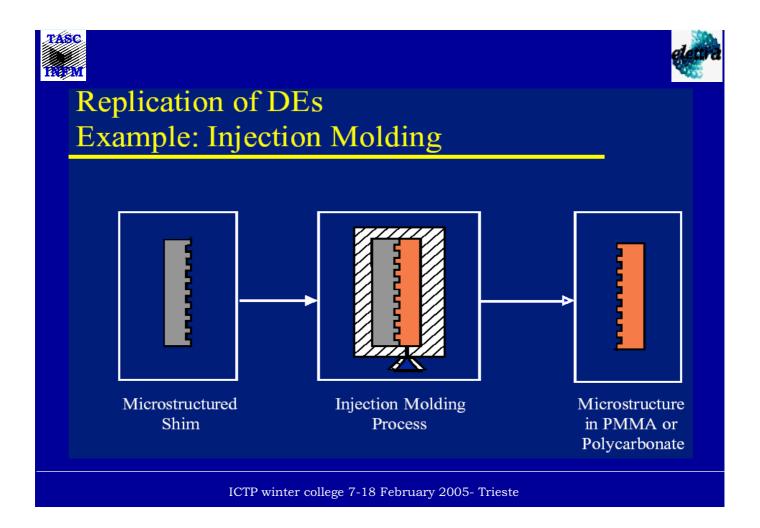
Continuous DE



Section of the designed 8-level structure of a beamsplitter



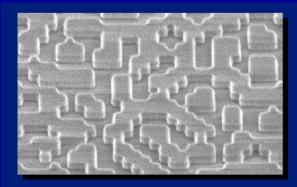
Section of the fabricated 8-level structure of a beamsplitter



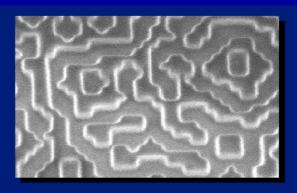


Replication Methods





Section of a binary quartzglass master



Section of replicated DE

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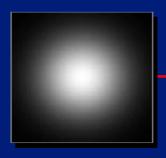




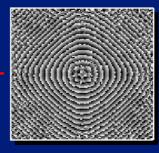
Implementation Example: Beamshaping (technical analysis)

Square-line beamshaper:

Output beam shape as specified Optical efficiency 86 % (theoretical)

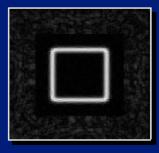


Illumination wave



far field

16-level DE



Output wave





Implementation Example: Ringfocus (technical analysis)

Circle-line beamshaper:

Output beam shape as specified

Optical efficiency greater than 85 % (theoretical)



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Implementation Example: Beamsplitter (fabricated)

1 to 40 ring beamsplitter:

Deflection angle of each beam 15 °

Optical efficiency 60 %; uniformity of output beams

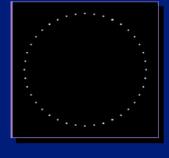


Illumination wave



far field

2-level DE

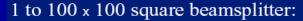


Output wave





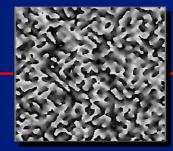
Implementation Example: Beamsplitter (technical analysis)



Optical efficiency 75% (theoretical) Uniformity of output beams

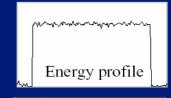


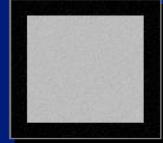




far field

8-level DE





Output wave: 100 x 100 light dots

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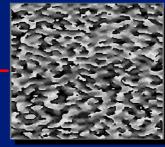
Example: digital Hologram of Tutenchamun (fabricated)

Digital hologram:

Output wave: image of Tutenchamun Minimum stray light



Illumination wave



8-level DE

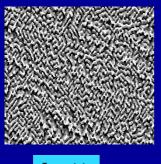






DOE's design problem:

find the phase function, $\Phi_{\rm DOE}(x)$, so that intensity distribution of the diffracted field, $I_{\sigma}=|E_{\sigma}|^2\sim I_{\sigma}$ (the desired intensity distribution) inside SW







 $\Phi_{\text{DOE}}(\mathbf{x})$

Design approaches:

- ray traycing
- iterative optimization techniques
- phase retrieval iterative algorithms

Io = $|\mathbf{Ao}|^2$ - **desired intensity distribution** Φ **o** - phase distribution **freedom degree**

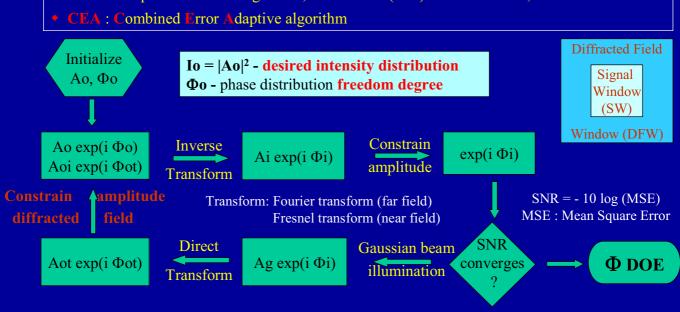
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DOE's design: Phase-retrieval iterative algorithms



- ERA: Error Reduction Algorithm, Aoi = Ao; DFW = SW
- AAA: Adaptive Additive Algorithm, Aoi = λ Ao+(1- λ)Aot inside SW; DFW > SW





Desired amplitude

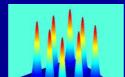
distribution in the

signal window SW

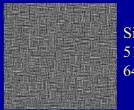
Computer simulation: Phase-retrieval algorithms



Planar array of optical tweezers

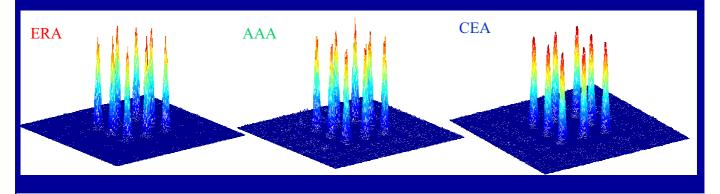


Phase DOE calculated with CEA



Size 1x1 mm 512x512 pixels 64 phase levels

Intensity distribution of the diffracted pattern obtained from DOE in the far field



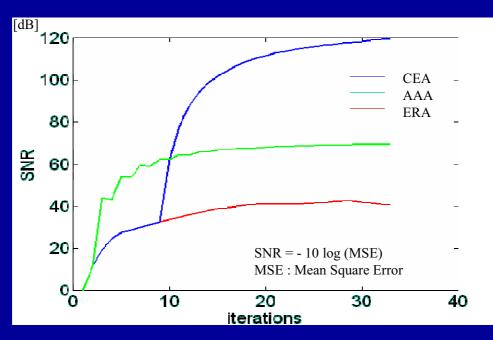
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Computer simulations: Phase-retrieval algorithms



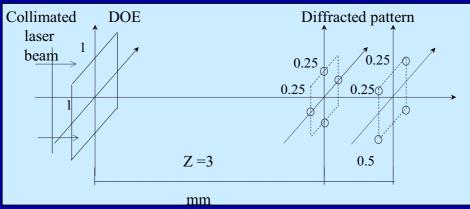
Comparison of the iterative algorithms in terms of SNR



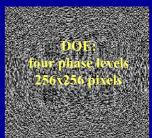


Computer simulation: CEA + Micro GA





- CEA: calculate the phase functions corresponding to the two planar arrays (40 iterations)
- Micro GA: calculate the optimum phase function that creates the 3D array (40 iterations)



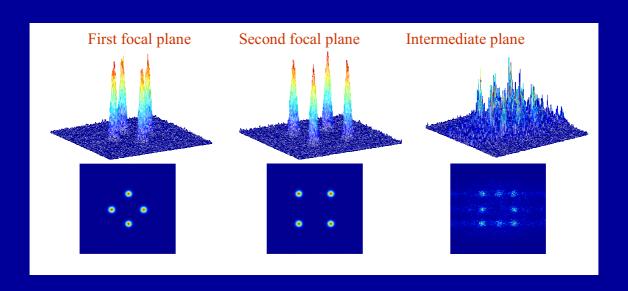
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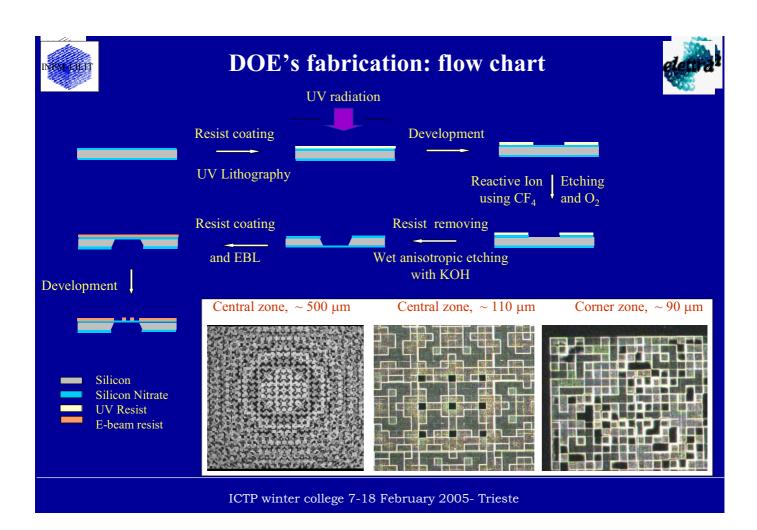


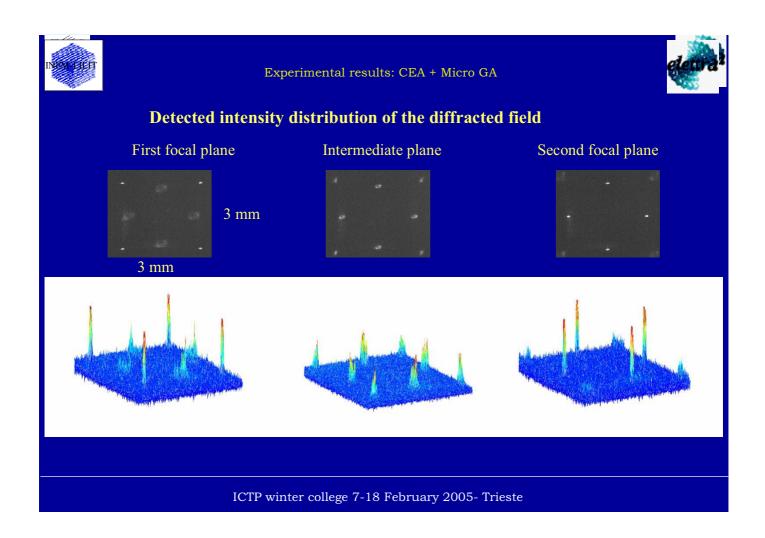
Computer simulation: CEA + Micro GA



Computed intensity distribution of the diffracted field

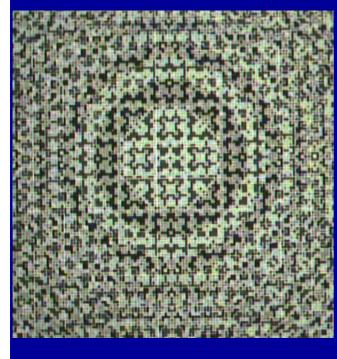












5x5 matrix for simultaneous Optical trapping



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Diffractive nanopptics for DIC x-ray microscopy





1. Contrast in X-ray microscopy

Refractive index in X-ray region: $n = 1 - \delta + i \cdot \beta$

 $\delta \rightarrow$ phase shift $\beta \rightarrow$ absorption

example: polyimide

photon energy [eV]	δ	β	δ/β	
100	2.3 ·10 ⁻²	4.9 ·10 ⁻³	4.7	
500	1.1 ·10 ⁻³	3.2 ·10 ⁻⁴	3.4	
5000	1.2 ·10 ⁻⁵	7.1 ·10 ⁻⁸	170	

→ phase sensitive techniques superior to absorption contrast!

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Motivation for microscopy techniques using phase information

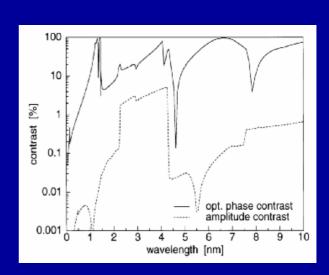


Absorption contrast: ~E -3

Phase contrast: ~E⁻¹

Use of phase shifting, real part of refractive index

- orders of magnitude higher contrast
- tremendous reduction of dose applied to object
- additional transmission information on low side of absorption edges (XANES, XRF!)



Amplitude and phase contrast for a model protein $C_{94}H_{139}N_{24}O_{31}$



General statements on DIC microscopy



Phase objects cannot be seen (difficult) when in focus with ordinary XRM (single ZP)

Phase objects retard or advance light that passes through them due to spatial variation in their refractive index and/or thickness

Needs for DIC microscopy

The image of DIC microscopes is formed from the interference of two mutually coherent waves with lateral displacements (shear) (of the order of the minimum size of the imaged structure and are phase-shifted relative to each other

Imaging characteristics

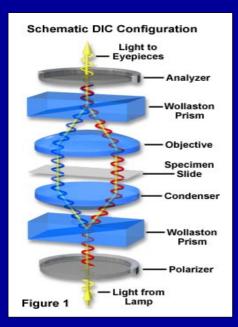
The intensity distribution in measured DIC images is given by a non linear function of the spatial gradient of a specimen's optical path length distribution along the direction of shear

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Differential Interference Contrast DIC



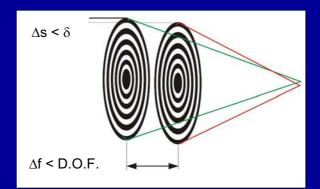


Visible light microscopy (Nomarsky set-up)

Shear of wave front division or distance of Airy disks in focal plane is smaller than optical resolution ("differential")

DIC for X-ray microscopy with ZPs:

- Distance Δf of both ZPs smaller than depth of focus
- Displacement Δs smaller than resolution δ



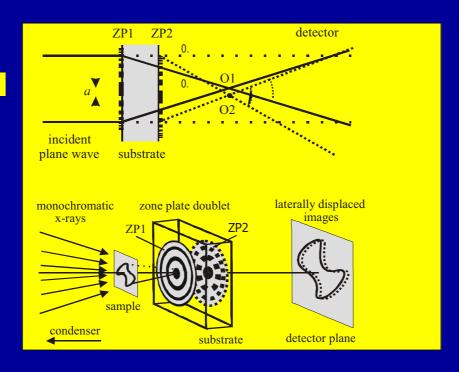


Differential Interference Contrast DIC



Working principle

TXM set-up



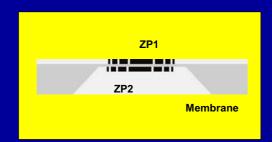
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ZP doublet for X-ray microscopy in DIC mode



First ZP doublet generated by E. Di Fabrizio and S. Cabrini



Technique: e-beam lithography and nanostructuring

Diameter: 75 μm
Outermost zone width: 200 nm
Focal length @ 4 keV: 50 mm

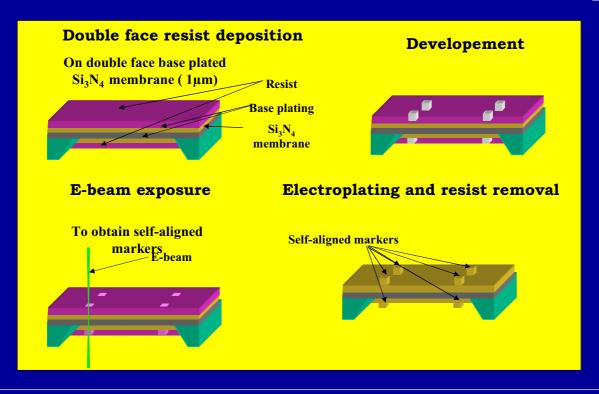
Efficiency: 10 %

Nominal displacement: 100 nm



Alignment markers definition



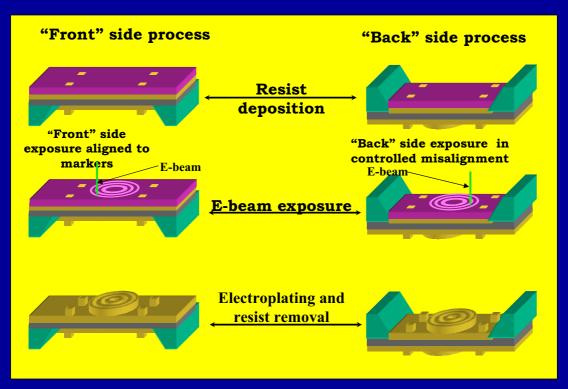


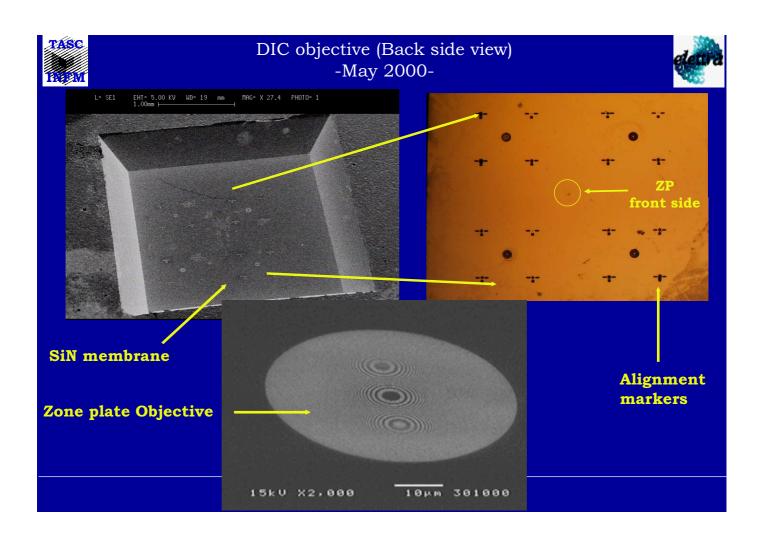
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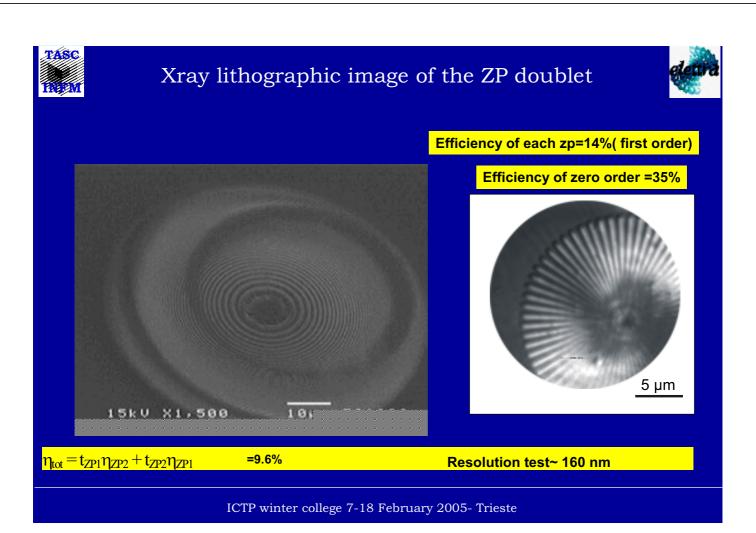


Double side ZP definition





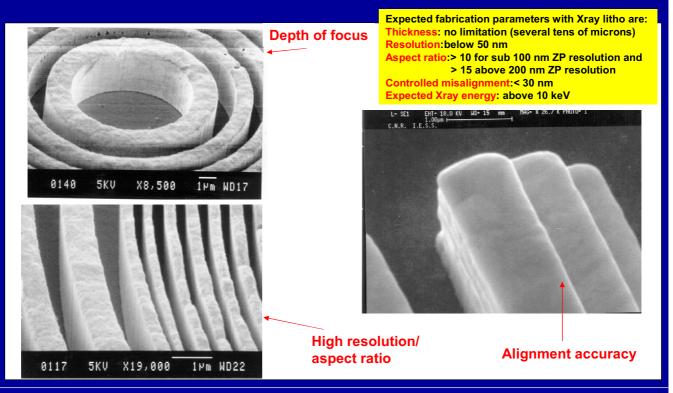






Potentiality of X-ray lithography for DIC fabrication for multi-keV energy



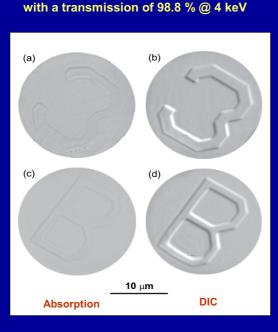


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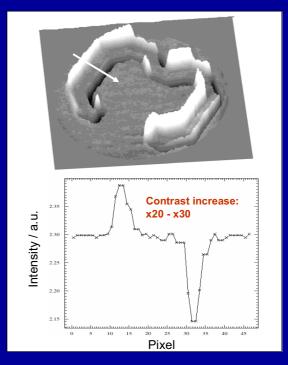


DIC X-ray microscopy with a full-field imaging microscope @ 4 keV





2 μm thick PMMA test structures

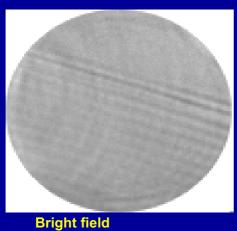


B. Kaulich, T. Wilhein, E. Di Fabrizio, S. Cabrini, F. Romanato, M. Altissimo, J. Susini (Nov 2000)

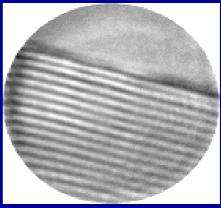


PMMA zone plate structure (250 nm) 2 micron thick:exposure time 10s

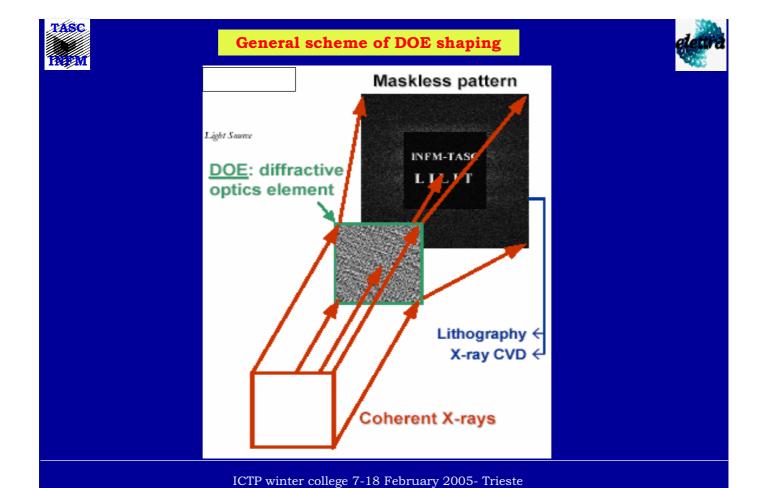




<u>5 μm</u>



X-DIC transmission





DOE's design



Numerical computation:

given a set of input data find the optimum output data which fit the requests

input data:

- •Source space: wavelength, size, geometry, intensity distribution
- •Image space: intensity or/and phase or/and polarization field distribution
- •DOE: size, resolution, material

output data:

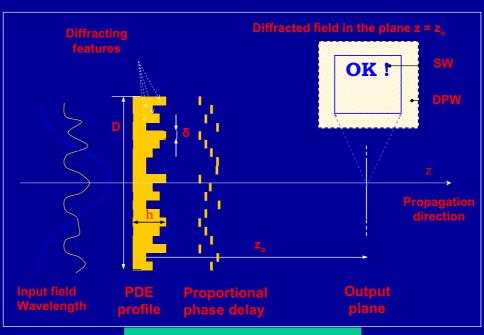
•DOE's phase or/and amplitude function

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Diffractive optical element scheme





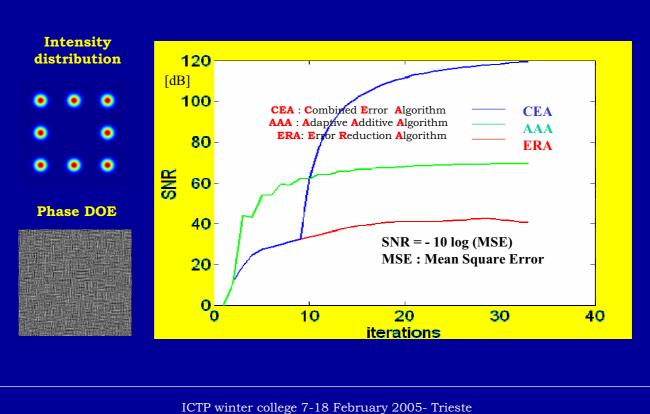
DOE's phase function:

 $\Phi_{\rm DOE}$ (x) = 2π (n-1) h(x) / λ



Comparison of the iterative algorithms

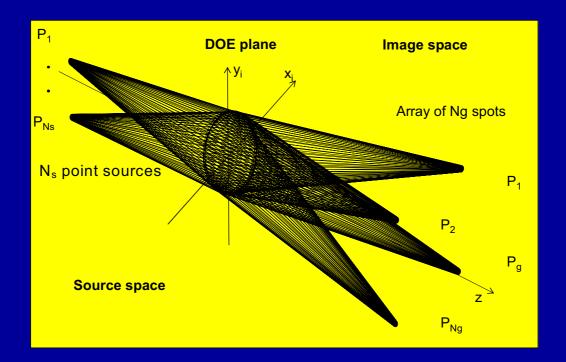






Spherical wave approximation





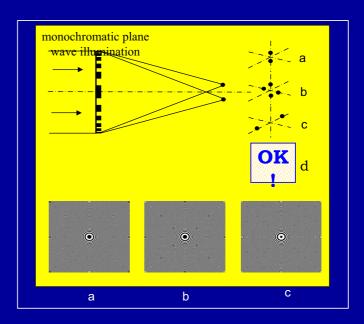


Development of multi-spot X-ray DOE



Optical scheme and calculated layout of 3 DOE's that generate 2 and 4 spots (a-b) on the same focal plane, and 2 spots along the same optical axis(c)

(d)Full beamshaping



The calculations are referred to a photon energy of 4 KeV and 5 cm focal length

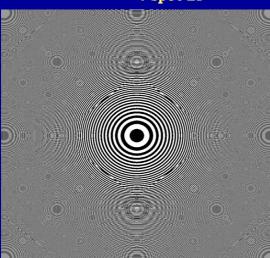
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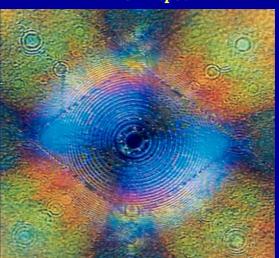
Development of multi-spot X-ray DOE



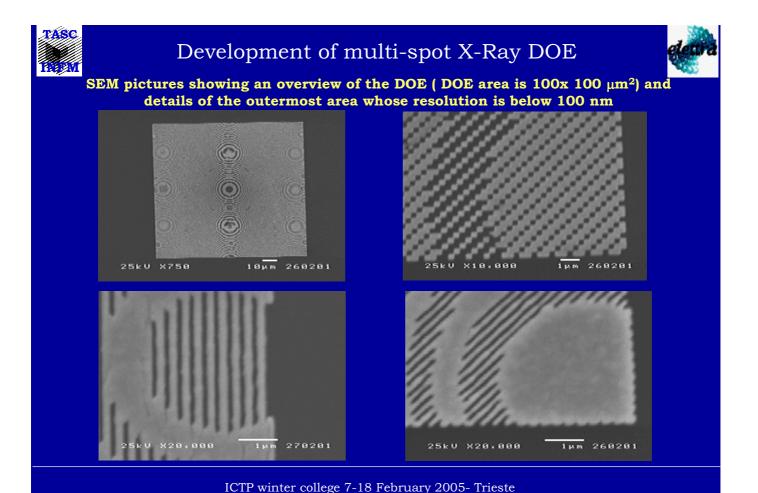
Calculated pattern of a 4 spot ZP

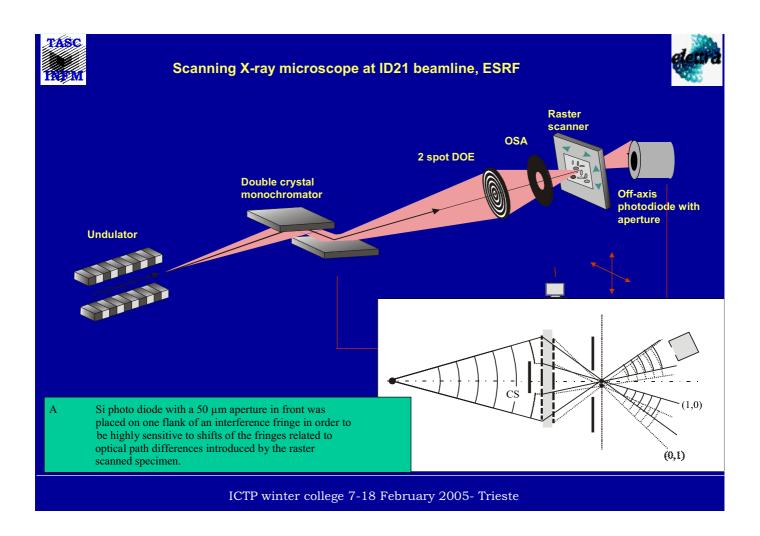


DIC visible light micrograph of 4-spot ZP



DOE size 0.1 x 0.1 mm2, pixel size: 100 nm, energy 4 keV designed and generated by the Lilit beamline group (2001)







2 spot DOE on SXTM at 4 KeV (February 2001)



2 μm thick PMMA test structures with a transmission of 99 % @ 4 keV

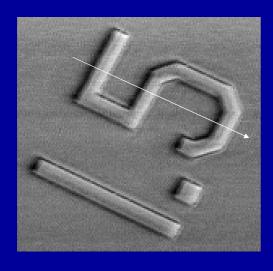


Image taken with the scanning X-ray microscope at the ID21 beamline, ESRF

Dwell time: 40ms / px with 200 x 200 px

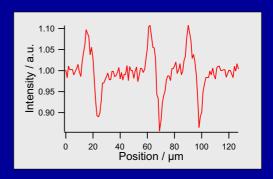


Image contrast: 25% in DIC

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Scanning transmission X-ray microscopy in DIC mode using a 2-spot ZP



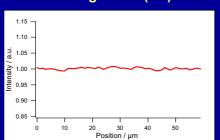


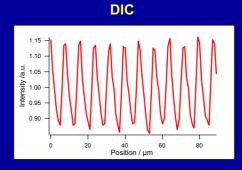
<u>2 μm</u>

2 μm thick grating structures in PMMA

4 keV

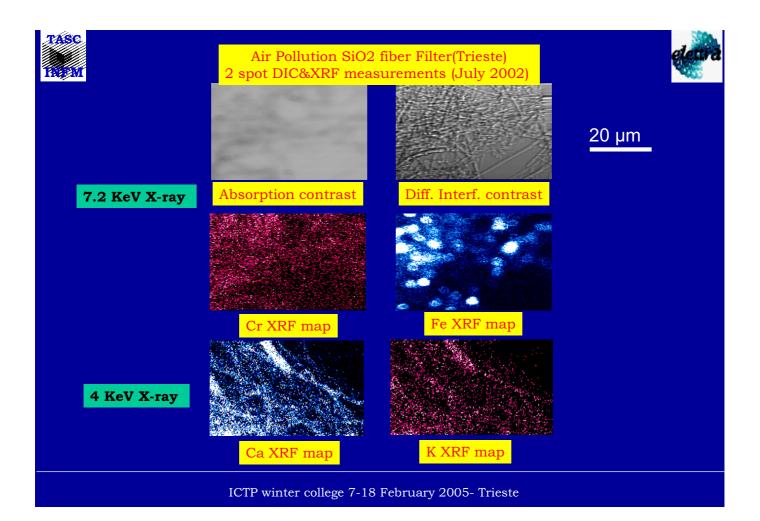
200 x 200 px 40 ms/px dwell

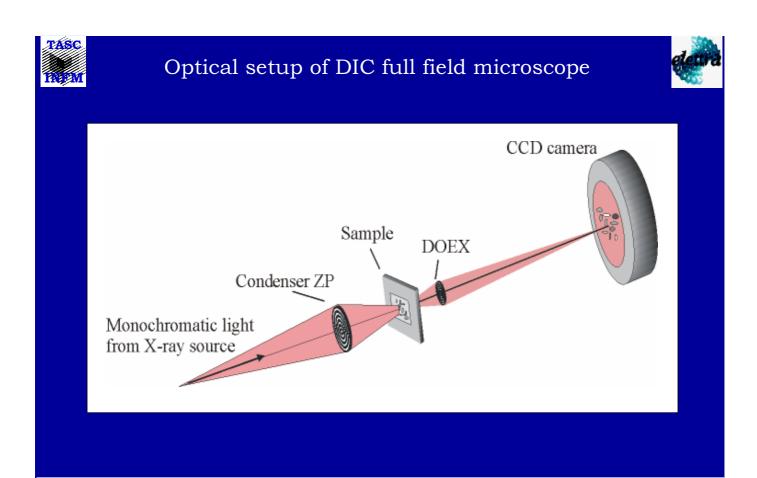




Contrast:

BF: 1 % DIC: 25 %



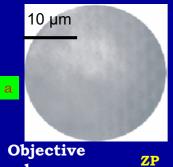




PMMA test structures (June 2001)



test structures(a=squares, b=toroids) 1 μm thick with a transmission of 99.99 % @ 4 keV



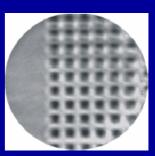
Objective lens:



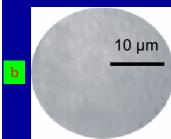
2 confocal spots DOE

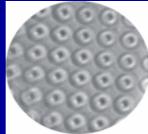


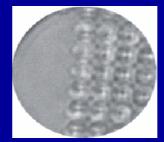
4 confocal spots DOE

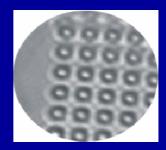


2 coaxial spots DOE







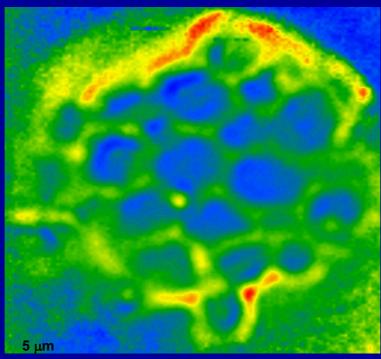


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Yeast cells: imaging in TXM at 4 keV by using diffractive optics

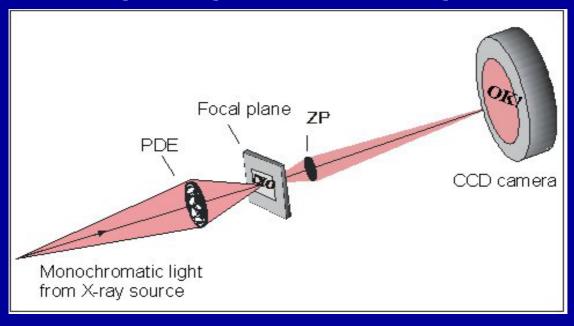








Complete X-ray- beam shaping Optical setup of DIC full field microscope

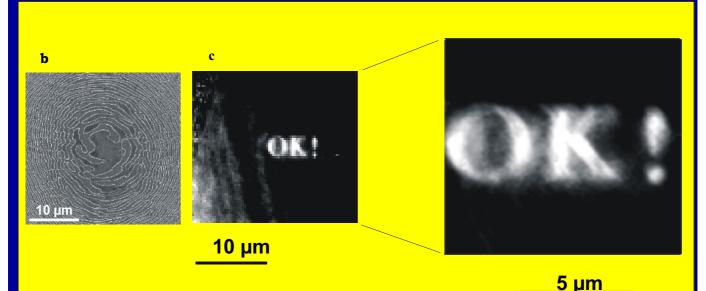


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Complete X-ray- beam shaping (June 2001)

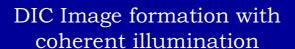




b - SEM image of the fabricated OK! DOE

c - The intermediate image formed by the DOE at the focal plane located at 50 mm is magnified 150 times on a CCD detector by a ZP that acts as an objective lens







$$i(\boldsymbol{x}) = a_1 \left| \int_{-\infty}^{+\infty} f(\boldsymbol{x_o}) \ h(\boldsymbol{x} \perp \boldsymbol{x_o}) \ d\boldsymbol{x_o} \right|^2$$

$$f(oldsymbol{x}) = \exp(-j\phi(oldsymbol{x}))$$
 Transmission function of the phase object

$$h(x,y) = 0.5 \exp(-j\Delta\theta)\delta(x - \Delta x, y) - 0.5 \exp(j\Delta\theta)\delta(x + \Delta x, y)$$

h(x,y) = Ideal PSF from ray-tracing (geometrical optics)

 Δx

=Shear in x direction

 $\Delta heta$

=constant phase bias

C. Preza et. al. J. Opt. Soc. Soc. Am. A/Vol. 16, No. 9. 1999

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Field Intensity in the DIC image



$$i(x, y) = a_1 \sin^2 \left[0.5 \left\{ \phi(x - \Delta x, y) - \phi(x + \Delta x, y) \right\} + \Delta \theta \right]$$

If the shear

 Δx

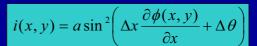
very small (differential) comapared to the size of the details of the specimen (or the resolution of the ZP) ==> the phase difference can be written as a phase gradient:

$$i(x,y) = a_1 \sin^2 \left(\Delta x \frac{\partial \phi(x,y)}{\partial x} + \Delta \theta \right)$$

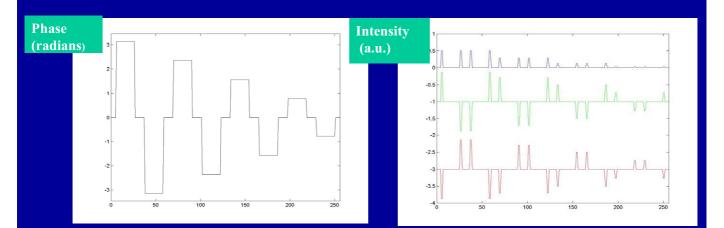
With DOE is possible to control the contrast through the bias $\Delta \Theta$



Bias retardation in DIC







- (a) The phase function of a one dimensional object described in 250 pixels;
- (b) Intensity distributions obtained with the same shear Δx = 2 but different bias values: $\Delta \theta = 0$ first line, $\Delta \theta = \pi/4$ second line, and $\Delta \theta = 3\pi/4$ third line; the signals are offset by 2 a.u. for a clear representation

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Spherical wave propagation approach

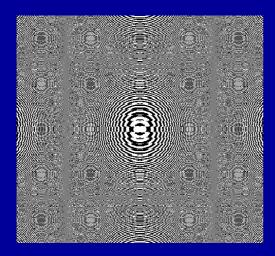


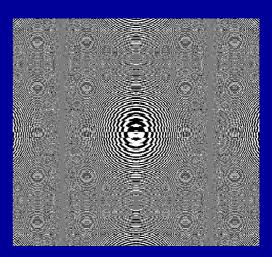
To calculate the phase function Φ_{PDE} , we assume that the light source which illuminates the PDE and the intensity distribution produced by the PDE can be described by point sources generating spherical waves $P_{\text{DE}} = \{\text{PDE} \mid \text{PDE} \mid \text{PDE}$





DOEs producing the same beam shearing (1 mm) but different bias: a) no bias, b) bias = π at 1 m from the DOE (DOE size= 2 cm, described in 480 pixels)



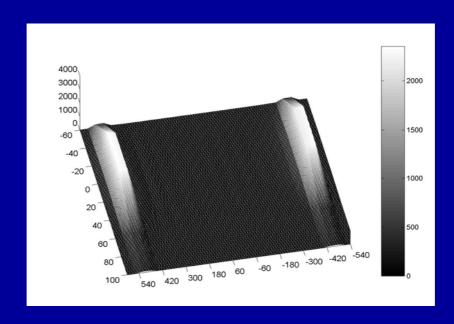


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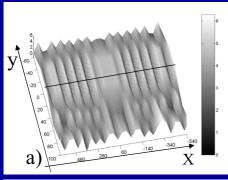


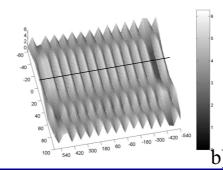
The intensity distribution obtained in the focal plane ($\Delta x=1 \text{ mm } \lambda=532 \text{ nm DOE}$ made by Hamamatsu SLM)

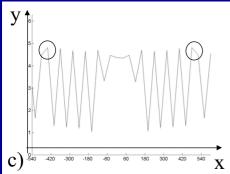


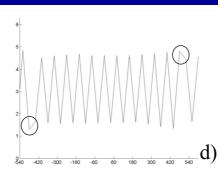












Phase distributions obtained in the focal plane of the DOE. For the 3D graphics (a,b) the phase is represented in radians on the z axis, the distances represented on x and y axes being expressed in microns. The phase distributions along the lines indicated in a) and b) are represented in the second line c) and d) clearly showing the presence of the π bias

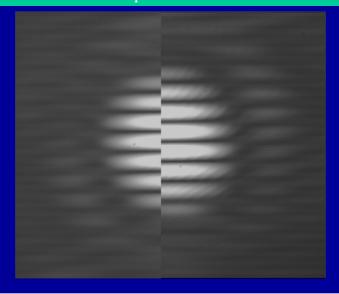
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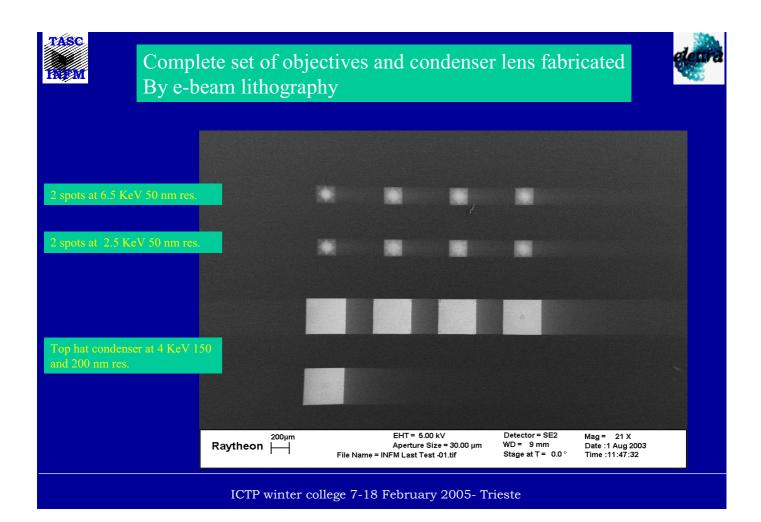


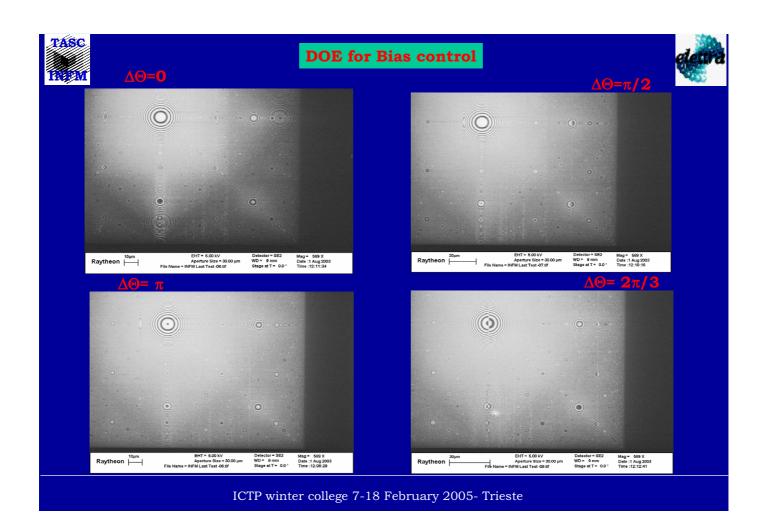
The interference patterns obtained after the focal plane of the DOEs implemented on the phase SLM Hamamatsu;

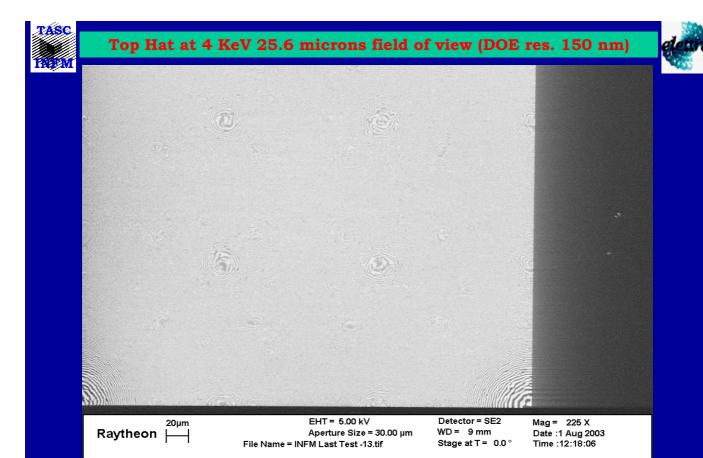
the left pattern corresponding to the DOE without bias is shifted with half of a fringe with respect to the right pattern which corresponds to the DOE with bias π

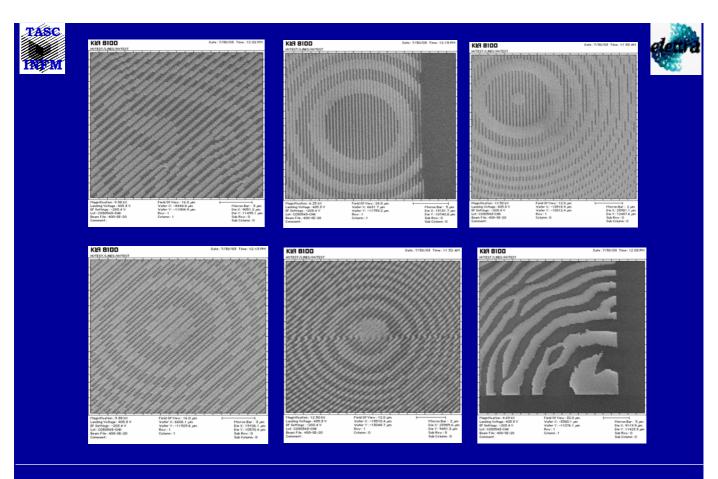


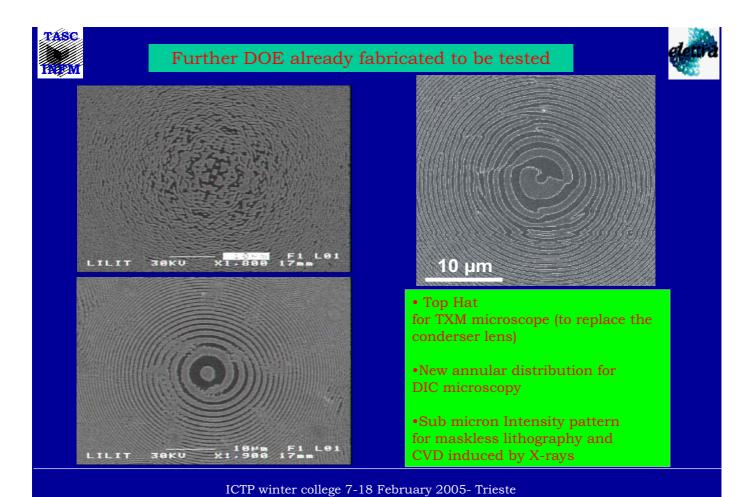
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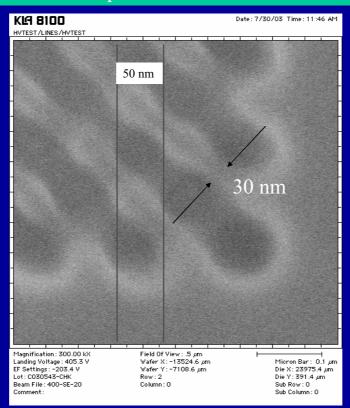






50 nm smaller pixel size with 30 nm details



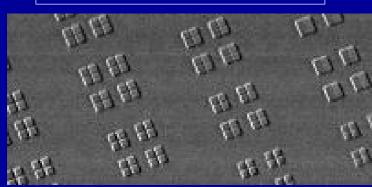




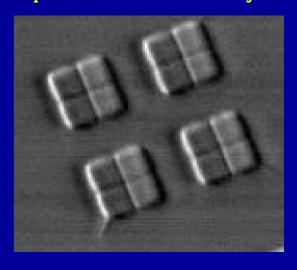
Experimental results DOE for XRM

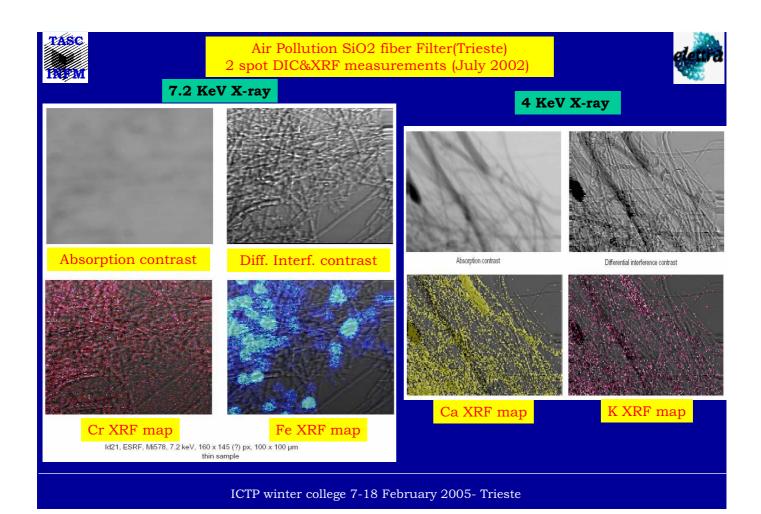


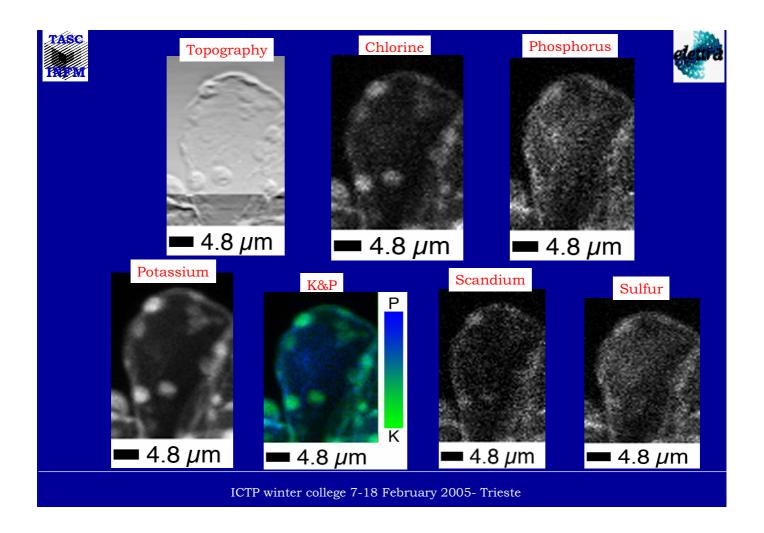
From the report
DOE Experiment
O. Dhez and M.Salom'e
7/10/2003

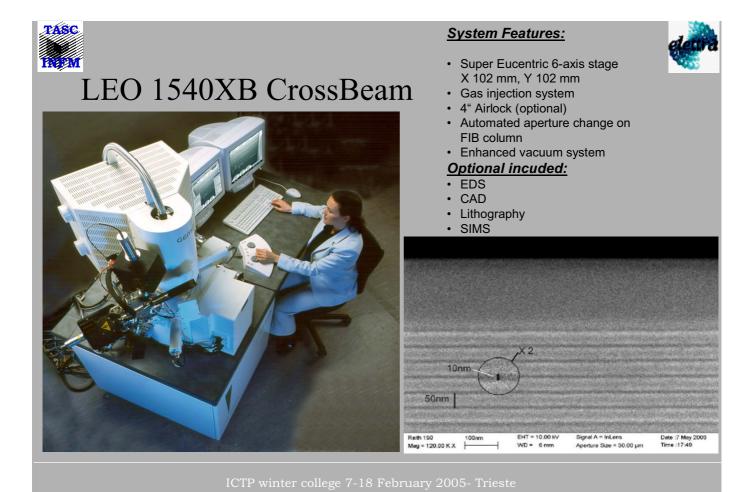


Experiment performed on ID21 SXM branch, in 16 bunch mode, under vacuum at 6.5keV. The sample used is the PMMA test object.







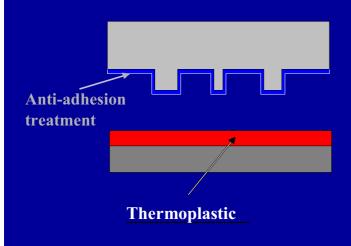




NanoImprinting



<u>Stamp materials</u>: Si, Si oxide, Si nitrate, Ni, W, quartz, sapphire





Embossing conditions

pressure: 50-100 bar

temperature: $T \sim T$ g^+ 90°

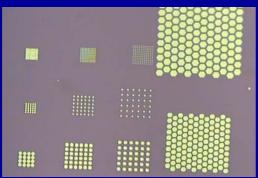
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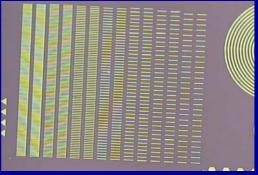


Patterning large features by Nanoimprint

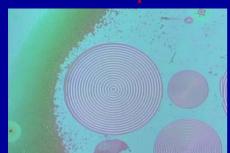




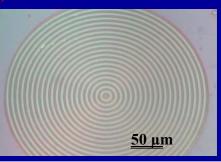




Imprinted structures: PMMA (plexiglass) on Silicon









Outlook and future investigations



- √ Two different DIC techniques using ZP doublet and X-Ray DOE
- ✓ Spatial resolution according to the design
- √ Technique has no limitation in spectral range as far as ZPs can be applied
- √ Full beam shaping achieved at X-ray wavelength

Future investigations:

- > Extension of experiments to soft and harder X-rays
- > Resolution limit toward state-of-art- fabrication technique
- > Theoretical investigations and simulations (transfer function)
- > Combination with spectro-microscopy for biological sample

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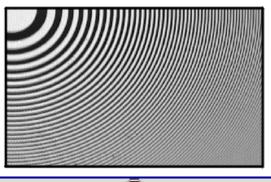
F. Polack, D. Joyeux, Lure& Universite Paris-Sud, Orsay, France

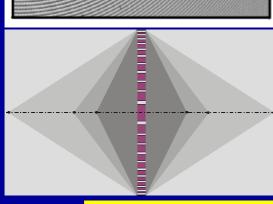
> Alexei Barinov, Maya Kiskinova ELETTRA



Basic properties of zone plates







Focusing, circular diffraction grating with radially increasing line density

$$\frac{1}{f} = \frac{1}{p} + \frac{1}{q} \qquad \text{if } n > 100$$

In terms of ZP parameters:

$$f_m = \frac{2}{m} D dr_m / \lambda$$

To avoid chromatic aberrations:

$$\frac{\mathsf{E}}{\Delta\mathsf{E}} \geq \mathsf{n} \, \mathsf{m}$$

$$\delta_{m} = \left[\left| \delta_{i,m}^{2} + \delta_{r}^{2} + \delta_{c}^{2} \right|^{1/2} = \left[\left| (1.22 \cdot dr_{n} / m)^{2} + \delta_{r}^{2} + (D \cdot \Delta E / E)^{2} \right|^{1/2} \right]$$

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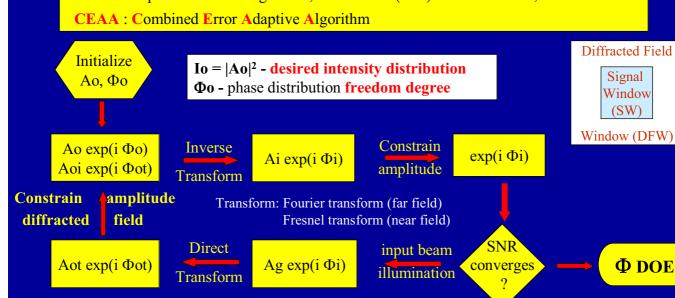


Phase – retrieval iterative algorithms



ERA: Error Reduction Algorithm, Aoi = Ao; DFW = SW

AAA: Adaptive Additive Algorithm, Aoi = λ Ao+(1- λ)Aot inside SW; DFW > SW



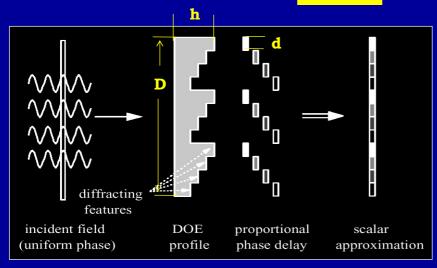


Scalar diffraction based model



scalar diffraction theory





DOE's phase function:

$$\Phi_{\text{DOE}}$$
 (x) = 2 π (n-1) h(x) / λ

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Coherence considerations



Spatial coherence (Van Cittert-Zernike)

$$D = 0.61 \quad \frac{L\lambda}{d/2} = 1.22 \quad \frac{f\lambda}{\delta}$$

- D: Diameter of coherently illuminated plane
- d: Source diameter
- L: Distance to observation plane

ZP shift
$$\Delta x < \delta = 1.22 \text{ dr}_N$$

Then:

If the separation(Δx) of the two superimposed images is below the resolution limit (δ) the two images will interfere without further restrictions to the spatial coherence of the source or, DIC works independent, of the spatial coherence of the illumination

Temporal coherence

$$I_{coh} = \frac{\lambda^2}{2 \Delta \lambda} > \frac{\lambda}{2} N = \Delta s_{max}$$

I_{coh}: coherence length
Δs_{max}: path length difference
N: Number of zones

$$\frac{\lambda}{\Delta \lambda}$$
 > N \approx N_{eff}= $\frac{(r + \Delta x)^2}{\lambda f}$

ZPs have to be treated to be in the same plane (within their depth of focus) (N>100)

Then:

No precautions to the source spectrum or other than for imaging with a single ZP



DIC X-ray microscopy with a full-field imaging microscope @ 4 keV







Spores of giant moss "Dawsonia superba"

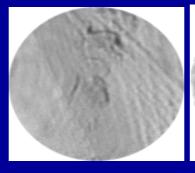
Exp. time: 20 s

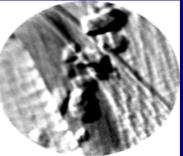
5 μm

Wing of a moss

Exp. time: 30 s

10 μm





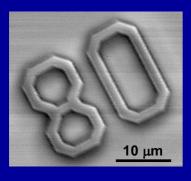
B. Kaulich, T. Wilhein, E. Di Fabrizio, S. Cabrini, F. Romanato, M. Altissimo, J. Susini (Nov 2000)

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Summary of DIC with ZP doublets

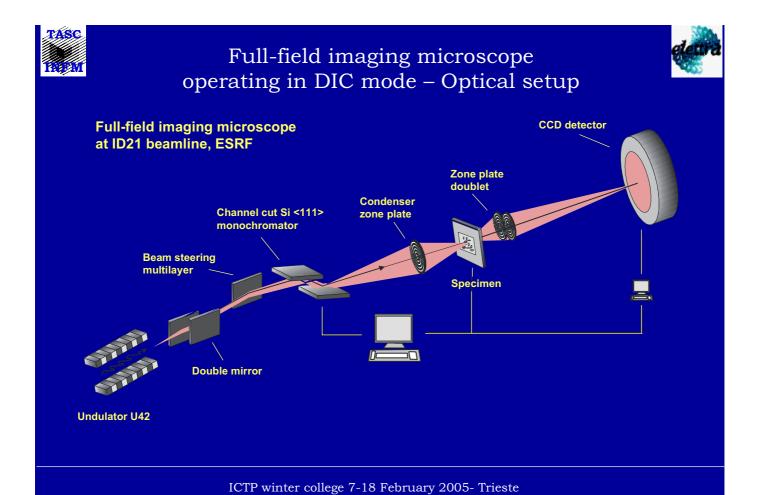


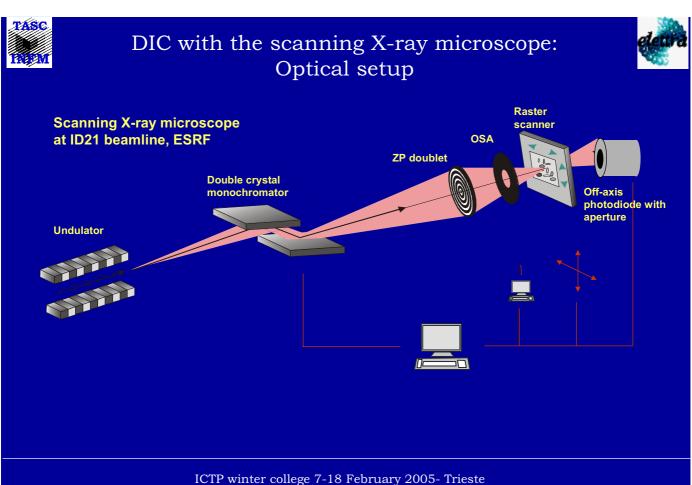


- DIC contrast technique, which is applicable for photon energies where ZPs work successfully (0.1 – 30 keV)
- Increase in image contrast of up to 20x 30x achieved
- Method can for the first time be applied in both transmission
 X-ray microscopy types (STXM and TXM)
- Alignment procedure not more complicated than for a single ZP

Improvements on ZP doublets fabrication:

- Simpler nanofabrication process (X-ray litho)
- •Optimization in ZP diffraction efficiency (10 % @ 4 keV meas.)
- Improvement in spatial resolution
- •higher energy range accessible > 10 keV

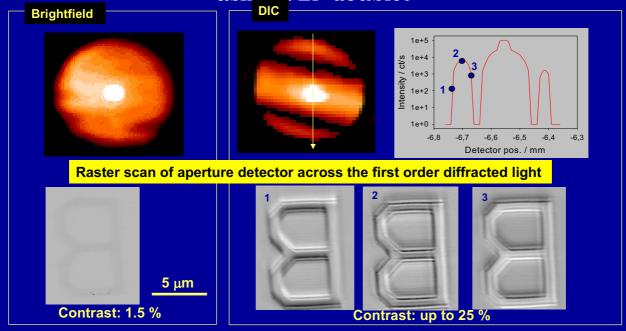






DIC with a scanning microscope (4 keV) using a ZP doublet





B. Kaulich, T. Wilhein, E. Di Fabrizio, F. Romanato, S. Cabrini, B. Fayard, J. Susini (Feb 2000) B. Kaulich, B. Fayard, U. Neuhaeusler, M. Salome, J. Susini (March 2000)

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Scalar versus Vectorial diffraction based models



Scalar based models

- •ray traycing, spherical wave propagation + superposition
- •phase retrieval iterative algorithms (PRIA)
- •global optimization methods: genetic algorithms, simulated annealing

Vectorial based models

- Finite element method (FEM)
- Boundary element method (BEM)
- Method of moments (MOM)
- Finite-difference method (FDM)
- Finite-difference time-domain (FDTD)



Propagation operators for scalar approximation



1 Fraunhofer (far field); propagation operator: Fourier transform

$$z > \pi D^2 / 4\lambda$$

2 Fresnel (near field); propagation operator: Fresnel transform

$$\sqrt[3]{\pi D^4 / 64\lambda} \le z \le \pi D^2 / 4\lambda$$

3 very near field; propagation operator: the Fresnel-Kirchoff integral

$$\lambda << z < \sqrt[3]{\pi D^4 / 64\lambda}$$

D 3 2 1 z

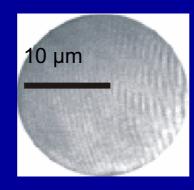
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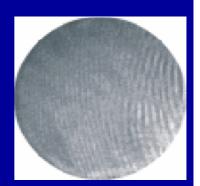


High resolution tests









Objective lens: 2 confocal spots DOE

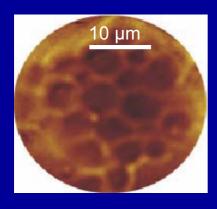
Sample: beamshaping DOE made by PMMA

Obs: the ultimate spatial resolution can be estimated being about 150 nm



Topography of biological samples @ 4 keV Measured with 2 coaxial spot DOE (June 2001)





array of yeast cells



frog blood cells



Iris flower fibers

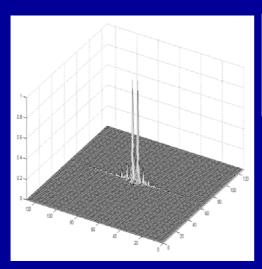
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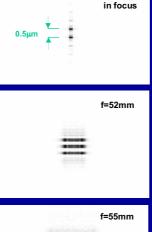
Simulation and measurements for multi-spot ZP

calculated

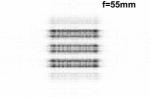


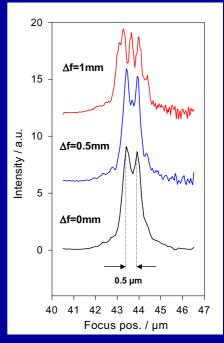


Simulation of 2-spot zone DOE in / out focal plane



f=50mm





Comp. Simulations by: E. Di Fabrizio et.al (Lilit) Measurements by: B. Kaulich, T. Wilhein, S. Cabrini, A. Barinov, J. Susini (Feb 2001)



Sum of oscillators



$$R = A[\cos \omega t + \cos(\omega t + \phi) + \cos(\omega t + 2\phi) + \cdots + \cos(\omega t + (n-1)\phi)],$$
(30.1)

$$A_R = A \frac{\sin n\phi/2}{\sin \phi/2}.$$

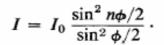




Fig. 30-5. The intensity pattern of a continuous line of oscillators has a single strong maximum and many weak "side lobes".

$$I = 4I_m \sin^2 \frac{1}{2} \Phi / \Phi^2.$$

are no higher-order maxima. If the scatterers are all in phase, we get a maximum in the direction $\theta_{\text{out}} = 0$, and a minimum when the distance Δ is equal to λ , just as for finite d and n. So we can even analyze a *continuous* distribution of scatterers or oscillators, by using integrals instead of summing.