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## Report Title

### Functional Organic Monolayers

#### ABSTRACT

1. New electron and X-ray diffraction methods have been developed for the structure analysis of organic sensors and detectors.
  2. The structure of an organic colorimetric biosensor has been determined by electron diffraction.
  3. A new hard hard light-element crystal structure has been discovered and its structure determined.
  4. A new method of lensless imaging for electrons and X-rays has been discovered. This method replaces lenses with a computer. X-ray diffraction patterns collected at the Lawrence Berkeley Laboratory Advanced Light source (synchrotron) have been inverted to images of test objects with a resolution of about 30nm.
  5. The lensless imaging method has been applied to electron diffraction patterns obtained in a TEM from metal nanoparticles containing defects. Atomic resolution images of these defects at interfaces have been reconstructed from electron microdiffraction patterns. The method makes it possible to reconstruct images from new forms of radiation for which no lenses exist.
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#### List of papers submitted or published that acknowledge ARO support during this reporting period. List the papers, including journal references, in the following categories:

##### (a) Papers published in peer-reviewed journals (N/A for none)

- 220 "Structure analysis of polymerized phospholipid bilayer by TED and Direct Methods. M. Stevens, M. Longo, D. Dorset, J. Spence. *Ultramic.* 90, 265 2002. DAAD190010500.
- 221 "Image reconstruction from electron and Xray diffraction patterns using iterative algorithms: experiments and simulations". U. Weierstall, Q. Chen, J. Spence, M. Howells, R. Panepucci and M. Isaacson. *Ultramic.* 90, 171 2002. ARO DAAD190010500.
237. "Holography of atoms". J.C.H.Spence. *Nature.* 410, p.1037. (2001).
- 241 Lensless Imaging. A Workshop on "New approaches to the Phase Problem for non-periodic objects" John C.H.Spence, M. Howells, L.D.Marks and J. Miao. *Ultramic.* 90, p.1-6 (2001). DAAD190010500
- 242 One-Step Synthesis and Crystal Structure of an Oligo(spiro-orthocarbonate) Matthew Braun,† David T. Vodak, Lycorgos Iordanidis, Jacques Plévert, Michael Stevens, Larry Beck, John C. H. Spence, Michael O'Keeffe, Omar M. Yaghi. *J. Am Chem Soc.* 124, 4942 2001.
- 246 Low-dose, low temperature CBED analysis of Aromatic Hydrocarbons. J. Wu and J.C.H.Spence. *Micros. and Microan.* 9, 428 2002. DAAD190010500
- 249 Phase recovery and lensless imaging by Iterative methods in optical and electron diffraction. J.C.H.Spence, U. Weierstall and M. Howells.. *Phil. Trans. (Thomas Young bi-centenary of two-slit experiment Special issue).* 360 875 2002. DAAD190010500
- 271 Solving non-periodic structures using Direct Methods: Phasing diffuse scattering. J.C.H. Spence, J. Wu, C. Giacobozzo, B.Carrozzini, G. L.Cascarano and H. Padmore. *Acta Cryst. A59*, 255-261. (2003).
- 282 Structural study of new hydrocarbon nano-crystals by energy-filtered electron diffraction J.S Wu, N. Melcer, W.P. Sharp, J.C.H. Spence, O.M. Yaghi. *Festchrift for F. Li. Ultramic.* 98, 145 2004.
- 284 "Structure and bonding in alpha-copper thalocyanine by electron diffraction". J. Wu and J.Spence. *Acta Cryst A59*, p.459. 2003.
- 288 Three-dimensional diffractive imaging for crystalline monolayers with one-dimensional compact support. J.C.H.Spence, U. Weierstall, K. Downing\*\* and R. M. Glaeser\*. *J. Struct. Biol.* 144, p209. 2003.
- 292 "A new thin-film phase of pentacene". J. Wu and J. Spence. *J. Appl Cryst.* In press. 2003.
- 293 "Tomographic diffractive imaging of monolayer crystals at atomic resolution with one-dimensional compact support". U. Weierstall, J. Spence and G. Hembree. *Optics . Express.* 11, p. 2335 (2003).
- 294 "Coherent x-ray diffraction imaging at the Advanced Light Source". Marchesini, Chapman, Hau-Riege, He, Howells, Kirz, London, Noy, Padmore, Rosen, Spence, Szoke, Weierstall. *Optics Express.* 11, p. 2344 (2003).
- 299 "Coherent Diffractive Imaging with X-rays and Electrons. J. Spence and M.R.Howells. *Synch. Rad. Instr. Conf. San Francisco*, 2003. *Am. Inst. Phys. Conf Proc.* 03.
- 308 Single Molecule Diffraction. J.C.H.Spence and B.Doak. *Physical Review Letters.* 92, 198102 (2004)
- 310 Interface structures in MgB2 thin films on (0001) SiC. J. Wu, N. Jiang, J.Spence. A. Pogrebnjakov, J.Redwing, X. Xi. *Appl Phys Letts* 85, 1155 (2004).
- 320 Iterative phase retrieval without support. J.Wu, U.Weierstall, J.Spence, C.Koch. *Optics Letters.* 29, p.1 2004.
- 321 Application of a modified Oszlanyi-Suto algorithm to experimental data. J.Wu, J.Spence, M.O'Keeffe, T.Groy. *Acta A60* 326. 2004.
- 322 Electron crystallography. The way forward. J.Spence. In *Nato ASI Series (2005) Erice 04. Summer school.*
- \* 1. J.S. Wu and J.C.H. Spence (2005): Diffractive electron imaging of nanoparticles on substrate. *Nature Materials.* In press. Nov. 05.
- \* 2. J.S. Wu, C.T. Koch and J.C.H. Spence (2005): Iterative phase retrieval for electron diffraction patterns from thin crystals. *Acta Cryst. A.* Submitted. 05
- \* 3. D. Starodub, B. Doak, K. Schmidt, U. Weierstall, J.S. Wu, G. Hembree J.C.H. Spence, M. Howells, M. Marcus, D. Shapiro, H. Chapman (2005). Damped and thermal motion of large, laser-aligned molecules in beams. *J. Chem. Phys.* In press. Nov 05
4. "An assessment of the resolution limitation due to radiation-damage in X-ray diffraction microscopy". M.Howells, T. Beetz, H. Chapman, C. Cui, J. Holton, C. Jacobsen, J. Kirz, E. Lima, S. Marchesini, H. Miao, D. Sayre, D. Shapiro, J. Spence. *J. Elec. Spectr. Rel Phenom.* (2004). In press. 05
5. J.S. Wu and J.C.H. Spence (2005): Phasing diffraction data from a stream of hydrated proteins. *J. Opt. Soc. Am.* 22, 1453-1459.

Number of Papers published in peer-reviewed journals: 25.00

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**(b) Papers published in non-peer-reviewed journals or in conference proceedings (N/A for none)**

Number of Papers published in non peer-reviewed journals: 0.00

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**(c) Papers presented at meetings, but not published in conference proceedings (N/A for none)**

Number of Papers not Published: 0.00

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**(d) Manuscripts**

Number of Manuscripts: 0.00

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Number of Inventions:

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**Graduate Students**

M. Stevens. 50%

Number of Graduate Students supported: 1.00

Total number of FTE graduate students: 1.00

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**Names of Post Doctorates**

Dr Jinsong Wu 100%

Number of Post Docs supported: 1.00

Total number of FTE Post Doctorates: 1.00

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**Names of Faculty Supported**

Prof J.C.H.Spence

Number of Faculty: 1.00

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**Names of Under Graduate students supported**

Number of under graduate students: 0.00

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**Names of Personnel receiving masters degrees**

**Number of Masters Awarded:** 0.00

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**Names of personnel receiving PHDs**

Dr. M. Stevens

**Number of PHDs awarded:** 1.00

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**Names of other research staff**

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**Sub Contractors (DD882)**

**Inventions (DD882)**

Under this ARO award 41512MS we have developed and applied an entirely new method of imaging atomic structures without using a lens. The method may therefore be used to form images with any radiation for which no lenses exist. Applications of this "lensless imaging" technique, which replaces a lens with a computer, have now been made using neutron beams, X-ray beams and electron beams. Only the scattering pattern is needed to form an image. This pattern of scattered intensity is applied to an iterative algorithm which solves the phase problem for scattering from isolated, non-periodic objects. We have now published examples at 30nm resolution using synchrotron radiation, and (Nature Materials 05) at atomic resolution using electron diffraction. The atomic resolution image shows atomic defects in the grain boundaries of a thin metal film. The resolution of these images is not limited by the numerical aperture of any lens, and the method shows high promise for application to solving proteins which cannot be crystallized. We have also extended the application of this phasing method to cryo-electron microscopy of two-dimensional protein crystals, where it allows three-dimensional image reconstruction using many diffraction patterns but few images.

Based on this solution to the non-crystallographic phase problem we have proposed (in Phys Rev Letts, 2004) a bold new method of solving protein structures which cannot be crystallised. (Crystallization is an important bottle-neck in new drug design and in structural biology generally). The method uses a stream of frozen droplets which run across a synchrotron beam, containing proteins. The proteins are aligned by a powerful infrared laser.

In addition to this work, we have developed new electron diffraction techniques to solve the atomic structure of the thin organic films used as sensors and detectors. We have also used these techniques to analyse a new very hard, light elements material whose discovery we collaborated in. This may be the first in a family of new, covalent carbonate network structures with useful properties (catalysis, coatings). Finally, using the same electron diffraction methods, we have discovered a new phase of pentacene, a material important for attempts to develop organic semiconductors.

During the course of this research we have developed a method of high aspect-ratio lithography, using the etched tracks from high-energy particle beams.

Details of all this work are contained in the publications listed earlier.