Microfocus/Polycapillary-Optic Crystallographic X-Ray System

This system generates an intense, nearly collimated beam suitable for crystallography.

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A system that generates an intense, nearly collimated, nearly monochromatic, small-diameter x-ray beam has been developed for use in macromolecular crystallography. A conventional x-ray system for macromolecular crystallography includes a rotating-anode x-ray source, which is massive (>500 kg), large (approximately 2 by 2 by 1 m), and power-hungry (between 2 and 18 kW). In contrast, the present system generates a beam of the required brightness from a microfocus source, which is small and light enough to be mounted on a laboratory bench, and operates at a power level of only tens of watts.

The figure schematically depicts the system as configured for observing x-ray diffraction from a macromolecular crystal. In addition to the microfocus x-ray source, the system includes a polycapillary optic — a monolithic block (typically a bundle of fused glass tubes) that contains thousands of straight or gently curved capillary channels, along which x-rays propagate with multiple reflections. This particular polycapillary optic is configured to act as a collimator; the x-ray beam that emerges from its output face consists of quasi-parallel subbeams with a small angular divergence and a diameter comparable to the size of a crystal to be studied. The gap between the microfocus x-ray source and the input face of the polycapillary optic is chosen consistently with the focal length of the polycapillary optic and the need to maximize the solid angle subtended by the optic in order to maxi-
mize the collimated x-ray flux. The spectrum from the source contains a significant component of Cu Kα (photon energy is 8.08 keV) radiation. The beam is monochromatized (for Cu Kα) by a nickel filter 10 μm thick.

In a test, this system was operated at a power of 40 W (current of 897 μA at an accelerating potential of 45 kV), with an anode x-ray spot size of 41±2 μm. Also tested, in order to provide a standard for comparison, was a commercial rotating-anode x-ray crystallographic system with a pyrolytic graphite monochromator and a 250-μm pinhole collimator, operating at a power of 3.15 kW (current of 70 mA at an accelerating potential of 45 kV). The flux of collimated Cu Kα radiation in this system was found to be approximately 16 times that in the rotating-anode system. Data on x-ray diffraction from crystals of tetragonal form of lysozyme (protein) in this system were found to be of high quality and to be reducible by use of standard crystallographic software.

This work was done by Marshall Joy of Marshall Space Flight Center, Mikhail Gubarev of the National Research Council, and Ewa Ciszak of Universities Space Research Association.

This invention is owned by NASA, and a patent application has been filed. For further information, contact Jim Dowdy, MSFC Commercialization Assistance Lead, at jim.dowdy@nasa.gov. Refer to MFS-31499.

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**Depth-Penetrating Luminescence Thermography of Thermal-Barrier Coatings**

**Temperatures at depths can be measured by luminescence decay of suitable thermographic phosphors.**

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A thermographic method has been developed for measuring temperatures at predetermined depths within dielectric material layers — especially thermal-barrier coatings. This method will help satisfy a need for noncontact measurement of through-the-thickness temperature gradients for evaluating the effectiveness of thermal-barrier coatings designed to prevent overheating of turbine blades, combustor liners, and other engine parts.

Heretofore, thermography has been limited to measurement of surface and near-surface temperatures. In the thermographic method that is the immediate predecessor of the present method, a thermographic phosphor is applied to the outer surface of a thermal barrier coating, luminescence in the phosphor is excited by illuminating the phosphor at a suitable wavelength, and either the time dependence of the intensity of luminescence or the intensities of luminescence spectral lines is measured. Then an emissivity-independent surface-temperature value is computed by use of either the known temperature dependence of the luminescence decay time or the known temperature dependence of ratios between intensities of selected luminescence spectral lines. Until now, depth-penetrating measurements have not been possible because light of the wavelengths needed to excite phosphors could not penetrate thermal-barrier coating materials to useful depths.

In the present method as in the method described above, one exploits the temperature dependence of luminescence decay time. In this case, the phosphor is incorporated into the thermal-barrier coat at the depth at which temperature is to be measured. To be suitable for use in this method, a phosphor must (1) exhibit a temperature dependence of luminescence decay time in the desired range, (2) be thermochem-