



Surface-Enhanced X-Ray Fluorescence

XRF spectra can be enhanced by large factors.

NASA's Jet Propulsion Laboratory, Pasadena, California

Surface-enhanced x-ray fluorescence (SEn-XRF) spectroscopy is a form of surface-enhanced spectroscopy that was conceived as a means of obtaining greater sensitivity in x-ray fluorescence (XRF) spectroscopy. As such, SEn-XRF spectroscopy joins the ranks of such other, longer-wavelength surface-enhanced spectroscopies as those based on surface-enhanced Raman scattering (SERS), surface-enhanced resonance Raman scattering (SERRS), and surface-enhanced infrared Raman absorption (SEIRA), which have been described in previous *NASA Tech Briefs* articles.

XRF spectroscopy has been used in analytical chemistry for determining the elemental compositions of small samples. XRF spectroscopy is rapid and quantitative and has been applied to a variety of metal and mineralogical samples. The main drawback of XRF spectroscopy as practiced heretofore is that sensitivity has not been as high as required for some applications.

In SEn-XRF as in the other surface-enhanced spectroscopies, one exploits several interacting near-field phenomena, occurring on nanotextured surfaces, that give rise to local concentrations of incident far-field illumination. In this case, the far-field illumination comes from an x-ray source. Depending on the chemical composition and the geometry of a given nanotextured surface, these phenomena could include the lightning-rod effect (concentration of electric fields at the sharpest points on needlelike surface features), surface plasmon resonances, and grazing incidence geometric effects. In the far field, the observable effect of these phenomena is an increase in the intensity of the spectrum of interest — in this case, the x-ray fluorescence spectrum of chemical elements of interest that may be present within a surface layer at distances no more than a few nanometers from the surface.

In experiments, SEn-XRF was demonstrated on aluminum substrates, the surfaces of some of which had been randomly nanotextured (see Figure 1) by briefly etching them in hydrochloric

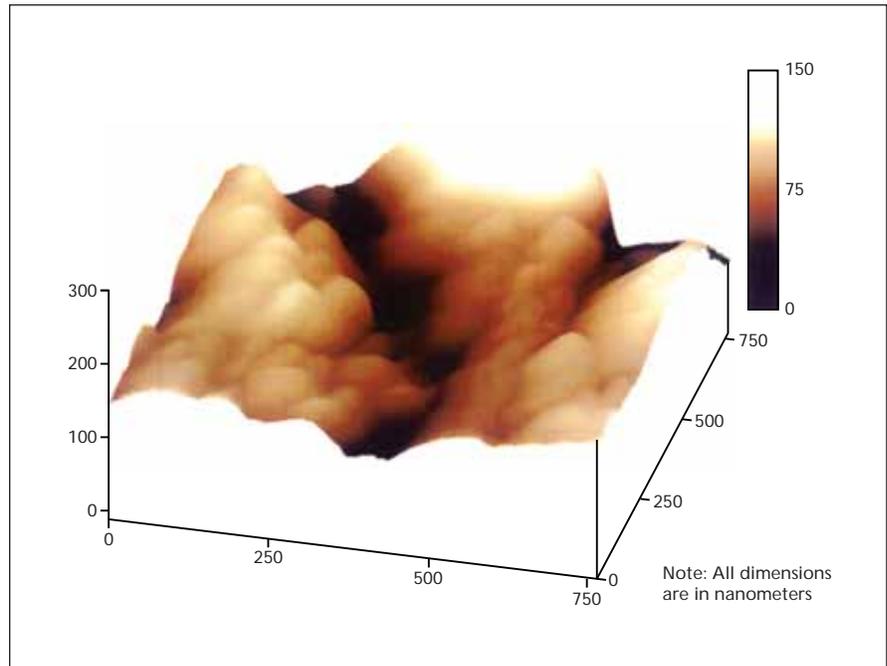


Figure 1. This Atomic-Force-Microscope Image shows a portion of the surface of an aluminum substrate that was roughened by a 1-minute etch in concentrated hydrochloric acid.

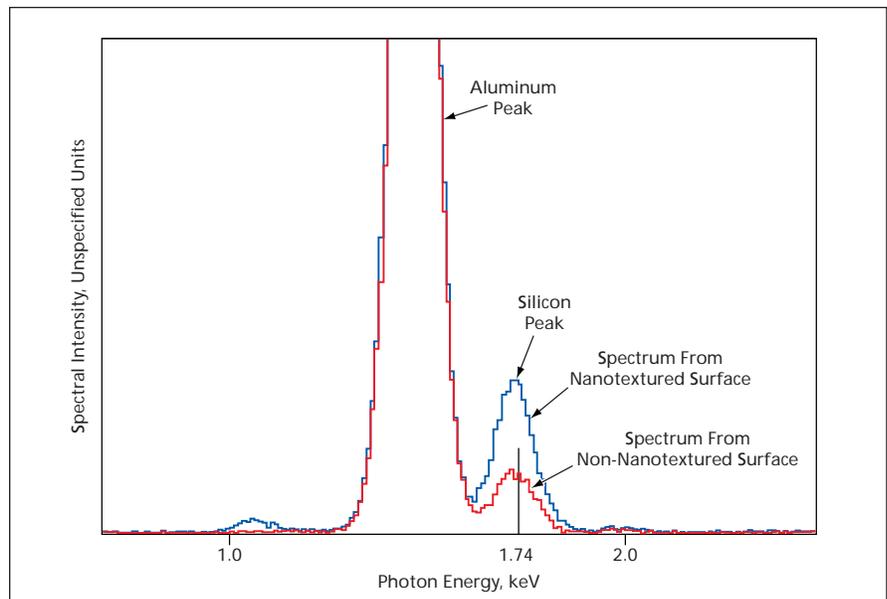


Figure 2. Silicon X-Ray Fluorescence Peaks at 1.74 keV were measured on aluminum surfaces coated with 340-nm-thick layers of silicone oil. The peak from the nanotextured surface was about 2.5 times as high as that from the nanotextured surface. Taking account of calculations showing that the enhancement occurs only within the 5-nm-thick sublayer closest to the surface, it was estimated that if the surfaces were coated to only 5-nm thickness, the factor of enhancement would be at least 170.

acid. Thin layers containing elements of interest (Si, I, K, Hg, and Pb) were deposited on the substrate, variously, from dilute salt solutions (in the case of K, Hg, and Pb), by vapor sublimation (in the case of I), or in a thin film of silicone oil (in the case of Si). XRF spectra of the thus-coated substrate surfaces were obtained by use of a commercial XRF microprobe instrument. In some cases, the XRF spectra of elements of interest on the nanotextured substrates were found

to be enhanced significantly over the spectra from the corresponding substrates that had not been nanotextured (for example, see Figure 2).

This work was done by Mark Anderson of Caltech for NASA's Jet Propulsion Laboratory. Further information is contained in a TSP (see page 1).

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Infrared Sensor on Unmanned Aircraft Transmits Time-Critical Wildfire Data

The sensor detects light at visible, infrared, and thermal wavelengths.

Dryden Flight Research Center, Edwards, California

Since 2006, NASA's Dryden Flight Research Center (DFRC) and Ames Research Center have been perfecting and demonstrating a new capability for geolocation of wildfires and the real-time delivery of data to firefighters. Managed for the Western States Fire Mission, the Ames-developed Autonomous Modular Scanner (AMS), mounted beneath a wing of DFRC's MQ-9 Ikhana remotely piloted aircraft, contains an infrared sensor capable of discriminating temperatures within 0.5 °F (≈ 0.3 °C), up to 1,000 °F (≈ 540 °C).

The AMS operates like a digital camera with specialized filters to detect light energy at visible, infrared, and thermal wavelengths. By placing the AMS aboard unmanned aircraft, one can gather in-

formation and imaging for thousands of square miles, and provide critical information about the location, size, and terrain around fires to commanders in the field. In the hands of operational agencies, the benefits of this NASA research and development effort can support nationwide wildfire fighting efforts. The sensor also provides data for post-burn and vegetation regrowth analyses.

The MQ-9 Unmanned Aircraft System (UAS), a version of the Predator-B, can operate over long distances, staying aloft for over 24 hours, and controlled via a satellite-linked command and control system. This same link is used to deliver the fire location data directly to fire incident commanders, in less than 10 minutes from the time of overflight. In the

current method, similarly equipped short-duration manned aircraft, with limited endurance and range, must land, hand-carry, and process data, and then deliver information to the firefighters, sometimes taking several hours in the process. Meanwhile, many fires would have moved over great distances and changed direction. Speed is critical. The fire incident commanders must assess a very dynamic situation, and task resources such as people, ground equipment, and retardant-dropping aircraft, often in mountainous terrain obscured by dense smoke.

*This work was done by Mark Pestana of Dryden Flight Research Center. Further information is contained in a TSP (see page 1).
DRC-010-020*

Slopes To Prevent Trapping of Bubbles in Microfluidic Channels

This idea helps to ensure functionality of micro-capillary electrophoresis devices.

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The idea of designing a microfluidic channel to slope upward along the direction of flow of the liquid in the channel has been conceived to help prevent trapping of gas bubbles in the channel. In the original application that gave rise to this idea, the microfluidic channels are parts of micro-capillary electrophoresis (microCE) devices undergoing development for use on Mars in detecting compounds indicative of life. It is necessary to prevent trapping of gas bubbles in these devices because unin-

terrupted liquid pathways are essential for sustaining the electrical conduction and flows that are essential for CE. The idea is also applicable to microfluidic devices that may be developed for similar terrestrial microCE biotechnological applications or other terrestrial applications in which trapping of bubbles in microfluidic channels cannot be tolerated.

A typical microCE device in the original application includes, among other things, multiple layers of borosilicate float glass wafers. Microfluidic channels

are formed in the wafers, typically by use of wet chemical etching. The figure presents a simplified cross section of part of such a device in which the CE channel is formed in the lowermost wafer (denoted the channel wafer) and, according to the present innovation, slopes upward into a via hole in another wafer (denoted the manifold wafer) lying immediately above the channel wafer. Another feature of the present innovation is that the via hole in the manifold wafer is made to taper to a wider