SURFACE ANALYSIS USING CONFOCAL RAMAN MICRO-SPECTROSCOPY

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1. INTRODUCTION

Recent developments in instrumentation and the availability of several high quality commercial Raman microscopes have made Raman micro-spectroscopy an emerging tool for surface analysis. Specifically, confocal Raman micro-spectroscopy allows for near diffraction-limited spatial resolution, with the chemical and structural information of vibrational spectroscopy. Modern single-dispersion spectrographs with notch filters, permitting excellent spectral resolution while minimizing Rayleigh interference and maximizing the detected signal, are also important developments.

The ability of Raman spectroscopy to probe molecular structure, coupled with the spatial resolution and light gathering power of a microscope, has created a powerful tool for surface analysis. This technique constitutes a non-destructive method of obtaining chemical and morphological information from a variety of different composite materials, surfaces and thin films. Since the introduction of Raman microscopy techniques, its application to materials research has increased. The ability of the microscope to obtain chemical information from regions as small as 1 μm² makes Raman microscopy complementary to other microanalysis techniques, such as electron microscopy or x-ray microanalysis. The high spatial resolution is a definite advantage over infrared microscopy, which provides similar chemical information but is limited by lower attainable resolution (~30 μm² for a conventional globar system and ~10 μm² for a synchrotron system).

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Metallization of Polymers 2.
2. INSTRUMENTATION

2.1 Conventional Raman Microscopy

Using a microscope for the collection optics of a Raman spectrometer helps solve one of the major limitations of Raman spectroscopy: the inherently low intensity Raman scattering effect. The invention of the laser, an ideal excitation source for Raman spectroscopy, catalyzed a renaissance in Raman spectroscopy. Replacing the mercury arc lamps previously used with far more intense laser excitation sources greatly increased the detectable Raman signal. Lasers are also highly monochromatic sources of radiation, which greatly simplifies the instrumentation. While the introduction of laser sources overcame the inherently poor signal intensity of Raman scattered light, the use of microscope objectives, with their large solid angle field-of-view and high degree of focusing, allowed for more of the scattered light in a given region to be captured and to reach the detector\textsuperscript{1,2}.

Microscope optical systems for Raman spectroscopy are directly linked to the non-intuitive observation that the Raman scatter intensity does not decrease with decreasing volume until the diffraction limit is approached\textsuperscript{2}; instead, scatter intensity remains constant. A significant problem with Raman microscopy is the power density incident on the sample; without significant attenuation, standard laser sources will destroy the sample in a very short time. While lower powered lasers are required, the increase in efficiency of the collection optics more than makes up for this. Microscopes use faster optical systems, focus more light onto the sample and collect more scattered light than conventional Raman spectrometers.

2.2 Confocal Raman microscopy

Confocal microscopy was introduced to overcome some of the limitations of optical microscopy. A point source at the diffraction limit is focused onto the sample and the enlarged image is analyzed through a pinhole\textsuperscript{3}. This technique allows much greater contrast and spatial resolution at the cost of reducing the image area down to a small part of the entire field-of-view. In order to capture the entire field-of-view, an XY-raster scan of the field can be performed, and a full 2-dimensional image is created through numerical analysis of the detector signals. Additionally, a 3-dimensional image can be created by optical sectioning, where the XY raster scans are performed sequentially with a Z-focus stage, and the resulting data cube is analyzed via numerical methods to recreate the optical image of the original object\textsuperscript{2}. 