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**The Investigation of Phase Evolution in Composite Ceramic  
Superconductors Using Raman Microscopy Techniques**

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# The Investigation of Phase Evolution in Composite Ceramic Superconductors Using Raman Microscopy Techniques

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Raman microspectroscopy and imaging techniques have been used to investigate key mechanistic features that influence the formation of layered Bi- and Tl-based superconducting phases during the thermal treatment employed to produce BSCCO and TBCCO composite conductors. Seminal information gained from these studies includes the location of lead-rich nonsuperconducting second phases (NSPs) and the identification of the constituent phases in certain NSP agglomerations that tend to resist dissipation as high-T<sub>c</sub> phase formation proceeds to completion.

## 1. INTRODUCTION

Raman microspectroscopy and imaging Raman microscopy offer unique opportunities for studying the evolution and spatial distribution of chemical phases in high-critical-temperature (high-T<sub>c</sub>) superconducting ceramics. When applied to compressed/sintered powders, silver-clad composite tapes/wires, and thin films in conjunction with powder x-ray diffraction, scanning electron microscopy, and energy dispersive x-ray spectroscopy, it is possible to gain seminal insights about the identity, size, shape, orientation, and spatial distribution of the various NSPs that form and dissipate during heat treatment of the BSCCO and TBCCO silver-clad composite tapes [1-3]. The results have permitted us to make specific chemical identifications of alkaline earth cuprate and lead-rich NSPs and to map their spatial distribution with respect to the layered superconducting phases.

## 2. EXPERIMENTAL

The experimental methods and instrumentation used in this research are reported elsewhere [1,2].

## 3. RESULTS AND DISCUSSION

Figure 1 presents an illustration of the type of measurement that can be made using Raman microspectroscopy and imaging Raman microscopy. The "defocused Raman spectrum" in Fig. 1 was obtained

by spreading the excitation laser over the circled area in the "white light image" of the specimen, which in this case is a transverse view of a fully-processed 19-filament Ag/Bi-2223 composite conductor. In the white light image shown in Fig. 1, the silver sheath region has been darkened by image processing to provide contrast with the superconducting filaments (three filaments are discernable in the image). From previous phase characterization studies [1-3] we know that the Raman features at 626, 570, and 520 cm<sup>-1</sup> are due to Bi-2223, (Ca,Sr)<sub>14</sub>Cu<sub>24</sub>O<sub>41</sub>, and (Ca,Sr)<sub>2</sub>CuO<sub>3</sub>, respectively. Using our imaging Raman microscope in the filter mode [1-2], we are able to determine the location within the circled area in the white light image from which the Raman scattering at each of the three frequencies is occurring as shown by the three circled Raman images to the right of the white light image in Fig. 1. Each of the three Raman images has been corrected to remove background (BG) effects [1-2]. In these three images the white regions represent the locations of the phases scattering at the frequency indicated to the right of each image. The image of the 626 cm<sup>-1</sup> feature, I(626)/BG, reveals that the layered Bi-2223 phase is located in bands along the silver sheath. The images for the 570 and 520 cm<sup>-1</sup> features show how the (Ca,Sr)<sub>14</sub>Cu<sub>24</sub>O<sub>41</sub> and (Ca,Sr)<sub>2</sub>CuO<sub>3</sub> NSPs tend to agglomerate in the center region of the filament. The appearance of over-lapping phase domains is associated with the penetration depth of the excitation laser. These types of measurements provide new

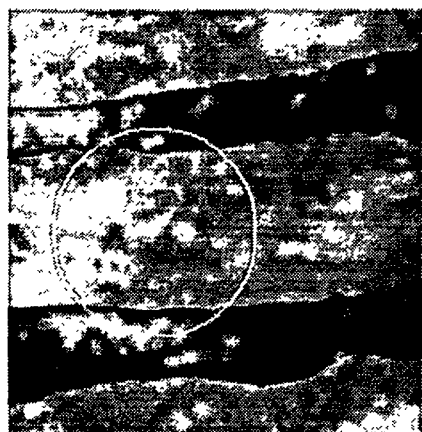
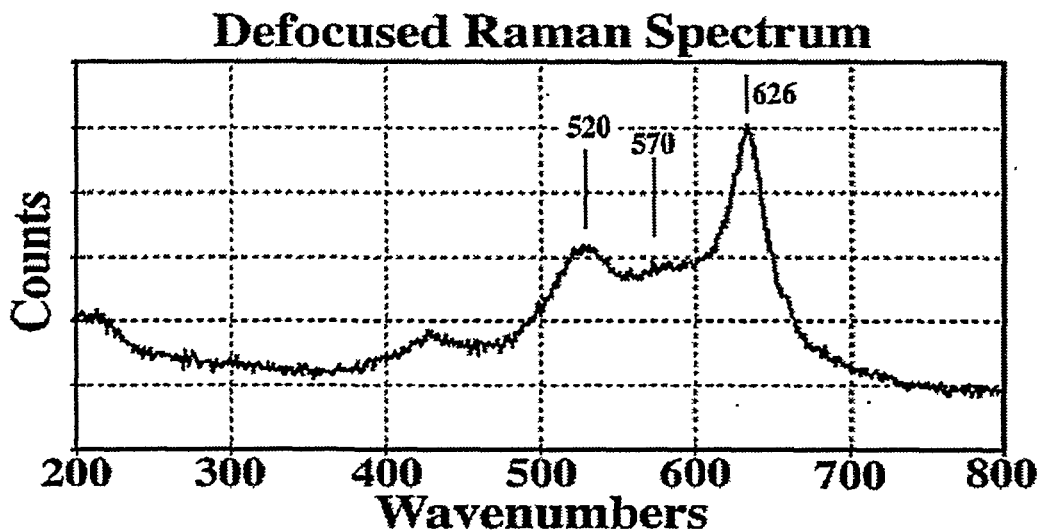
information about phase evolution and microstructure development during the synthesis/processing of cuprate-based ceramic superconductors.

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**White Light Image**

10 μm



**I(520)/BG  
2/1**



**I(570)/BG  
14/24**



**I(626)/BG  
Matrix**

$$BG = (I(480) + I(700)) / 2$$

Figure 1. Raman microscopy results for a fully-processed, 19-filament Ag/Bi-2223 composite.  
(See text for an explanation)