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RISE imaging of various phases of SiC in sintered silicon-carbide ceramics

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Silicon carbide (SiC) is a high-performance ceramic made of high quality, non-oxide powders which can be manufactured to the requirements of a wide range of applications. Ceramics made of silicon carbide play a key role as materials for high temperature applications due to their resistance against corrosion, heat, and wear. Furthermore, due to its wider band gap and resistance to radiation damage and electrical breakdown it is also an attractive material for the fabrication of microelectronic and optoelectronic devices. The properties of such ceramics strongly depend on the microstructure and grain growth of the polytypes of silicon carbide. In this contribution we studied recrystallized silicon carbide (RSiC), a pure silicon carbide material with 11-15% open porosity. This material is sintered at very high temperatures from 2,300 to 2,500° C, at which a mixture of extremely fine 4µm diameter grains (10%) and 90% coarse grains with a diameter of 100 µm is converted to a compact SiC matrix without shrinkage [1]. An overview SEM image of RSiC, acquired under high vacuum conditions at 1 kV acceleration voltage, is shown in Fig. 1. The pores appear as dark features in the SEM image and cover 20% of the analyzed surface. Large areas between the pores appear featureless, indicating a uniform composition of SiC. Fig. 2a shows the SEM image of an area between pores, where a very light contrast of grains become visible. This gives rise to study the same area also using high resolution confocal Raman imaging integrated in the vacuum chamber of the SEM [2,3]. Four different polytypes of SiC were detected on this sample area, shown in the overlay of the Raman and the SEM image, the RISE image in Fig. 2. Raman spectroscopy is sensitive to the nature of chemical bonding, revealing the structural differences in the RSiC ceramics, thus different crystallographic grains within the uniform area of the ceramics [4,5]. On the presented RISE-image the four SiC-phases correspond to: 6H-SiC (red), 15R-SiC (blue), 6H-SiC+4HSiC (green) and 6-H SiC with impurities (aqua). Impurities lead to shifts and broadening of the Raman band around 970 rel. 1/cm, associated with tensile (downshift) or compressive strain of the 6-H SiC [6]. The aim of this presentation is to reveal unique sub-structures of silicon carbide polytypes in RSiC-ceramics formed by recrystallization using correlative Raman-SEM microscopy, both techniques integrated within one instrument.

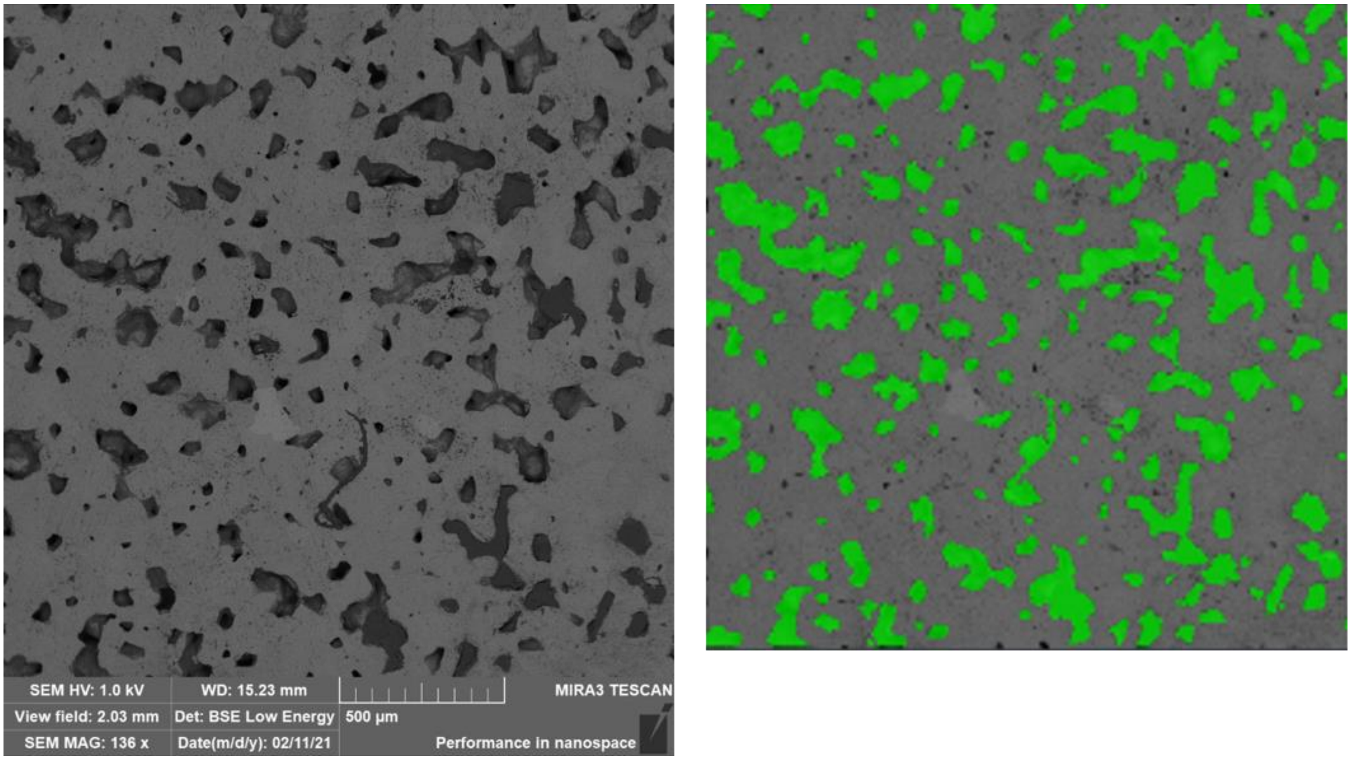

Figure 1. Microstructure of RSiC revealing the pore distribution in the ceramics. SEM image (left) and highlighted pores (right).

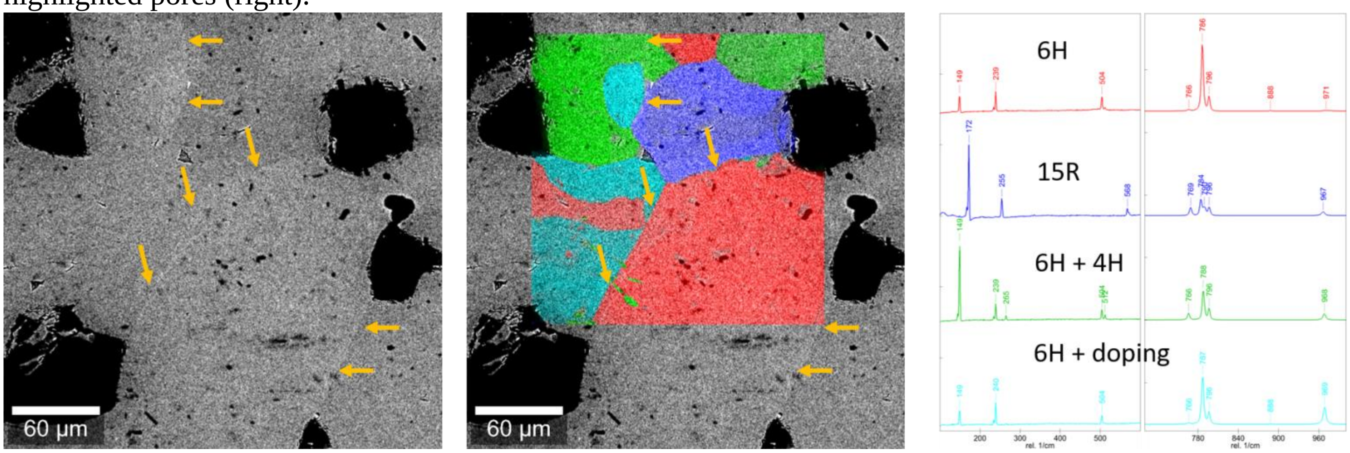

Figure 2. SEM images (left), RISE image (middle) and color-coded Raman spectra of polytypes of SiC (right) measured on the RSiC ceramics. SEM imaging parameter: low vacuum conditions, 15 kV acceleration voltage. Raman image parameters: 200x200µm2, 400x400 pixels, integration time: 0.05 ms, total acquisition time of 2,2 hours for 160 thousand spectra.

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