application note

Lise-Meitner-Straße 6, D-89081 Ulm, Germany
Tel. +49 (0) 731 140 700, Fax. +49 (0) 731 140 70200
www.witec.de, info@witec.de

Stress in Si
Combining Confocal Raman and Atomic Force Microscopy

Vickers Indent

The indentation hardness test as an element of the hardness measurement makes it possible to acquire a response curve for a material while measuring force and indentation depth. A special instrument, called a nanoindentor was developed for this test. With this instrument it is possible to produce very small indentations with a depth of a few microns and a force on the order of a few µN.

The advantage of this experiment is the localized deformation of the material that enables the examination at small volumes. For the indentation hardness test, a hard form - a pyramid (Vickers indenter) in our experiment - was pushed into the sample material with a controlled force and penetration.

In this study, the stress field around a Vickers indent was analyzed using the relative shift of the 520/cm Si-Raman line. The Vickers indent was performed with a force of 50 mN, resulting in a pyramidal hole of 210 nm depth and 2,75 µm diagonal size, as shown in the AFM image in fig. 1 (5 x 5 µm scale).
The images below show a comparision of the topography (10 x 10 µm, 5 nm vertical scale, fig. 2) around the Vickers indent and a stress image of the same area obtained in Raman Imaging Mode (10 x 10 µm scale, fig. 3), with which a complete Raman spectrum is acquired at every pixel, 100 x 100 pixels =10 000 spectra in this case. The Raman image is provided by simply rotating the turret from the AFM objective to a standard optical microscope objective (Nikon, 100 x air, NA = 0.9 NCG) without moving the sample. As can be seen, the extent of the stress field can be detected more than 5 µm from the center of the indent. The stress image was obtained with 10 mW excitation power (532 nm) and 70 ms integration time per spectrum.

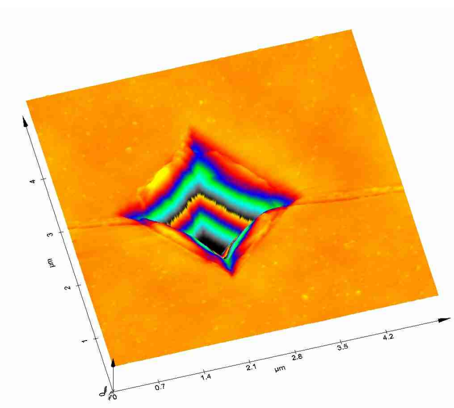


Fig. 1: AFM image, 5 x 5 µm scale.

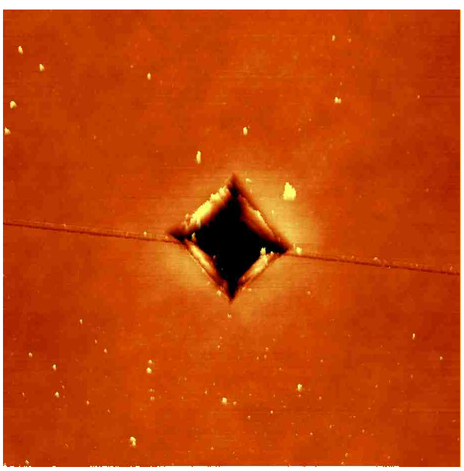


Fig. 2: AFM image: Topography around a Vickers indent,, 10 x 10 µm scale

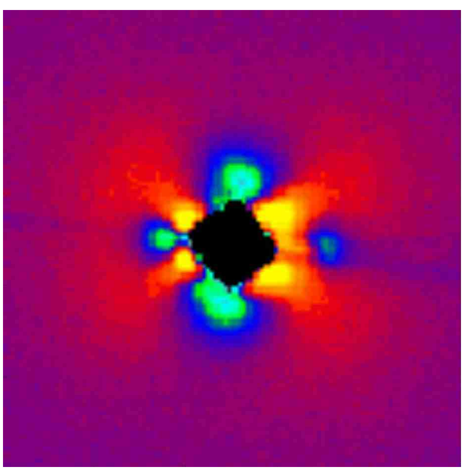
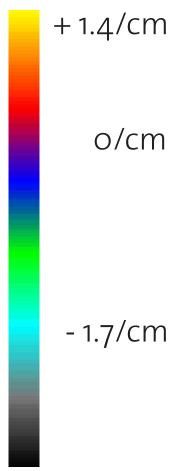


Fig. 3: Stress image of the same area as in fig. 2, obtained in the Raman Imaging Mode, 10 x 10 µm scale



Scale bar

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The positions of the three cross sections (labelled 1-3) are marked in the stress image in fig. 4 and displayed in fig. 5 to fig. 7.
As expected, the stress field has the symmetry of the Vickers pyramid. The tensile strain (blue in the stress image, fig.4) appears at the corners of the pyramid, while the compressive strain (yellow in the stress image, fig. 4) appears along the flat sides.
The cross sections show the extreme precision of the stress measurement with a standard deviation of only 0,021/cm. This accuracy was obtained by determining the peak position of the parabolic fit of the Si-Raman line in each measured spectrum. This degree of precision was achieved with an integration time of 70 ms per spectrum.
As the relation between peak shift and stress for silicon is 1 1/cm ($\Delta\omega$) = 435 MPa (σ), an accuracy of 0,02/cm corresponds to a stress sensitivity of only 9 MPa.

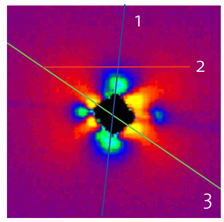

Fig. 4: Stress image, obtained in Raman Spectral Imaging Mode, 10 x 10 µm scale.

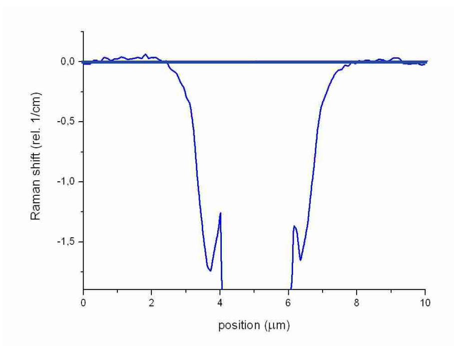

Fig. 5: Cross section, marked position 1 in fig. 4.

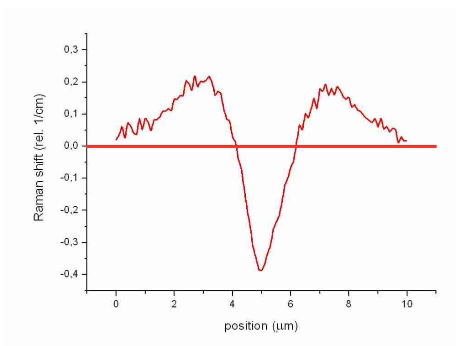

Fig. 6: Cross section, marked position 2 in fig. 4.

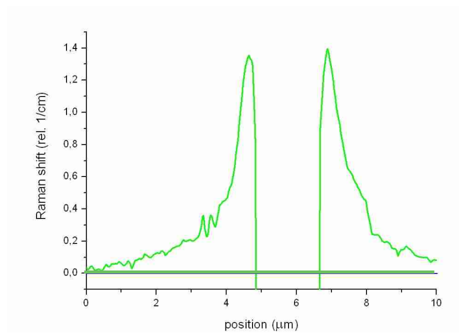

Fig. 7: Cross section, marked position 3 in fig. 4.

Samples courtesy of Helmut Fischer GmbH, Sindelfingen, Germany

Si/SiGe strained epitaxial film on silicon

The second sample is a 50nm SiGe layer on a silicon substrate with a 10nm cap layer of epitaxial silicon. The lateral homogeneity of strain in strained Si/SiGe materials is very important for the production of high mobility IC that use these materials. The SiGe layer has a certain lattice mismatch to pure Si which depends on the Ge concentration. This strain relaxes partially if the layer thickness exceeds the critical layer thickness. This critical thickness is not only a function of the Ge concentration, but also a function of process temperature and time. The lattice mismatch results in the formation of misfit and associated threading dislocations which lead to the characteristic crosshatch pattern typically seen on the strained Si epilayer surface.
An example of this crosshatch pattern is shown in the AFM image of Figure 8a. Scan range is 20 µm x 20 µm and the topography scale is 30nm. The scan is rotated by 45 degrees with respect to the <110> directions of the pattern. When performing a Raman analysis of this structure, the penetration depth of light into the structure has to be taken into account. For this excitation wavelength (532nm), a penetration depth of 760nm for pure silicon, and 540nm for $Si_{0.7}Ge_{0.3}$ can be calculated.
Therefore, the main contribution comes from the Si substrate. This is clearly visible in Figure 8c, which shows a single Raman spectrum obtained at this structure. The three peaks corresponding to the SiGe layer: the Ge-Ge (200 1/cm to 300 1/cm), Si-Ge (380 1/cm to 450 1/cm) and Si-Si (502 1/cm) stretching

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modes, are indicated, along with the Si-Si (520 1/cm) peak of the substrate. If the top silicon layer is to be analyzed, a shorter excitation wavelength with shallower penetration depth can be used.
The stress in the partially relaxed SiGe layer can be measured by analyzing the variation of the peak position of the Si-Si peak at 502 1/cm, which is shown in Figure 8b. Scan range is 20 µm x 20 µm with 100 x 100 = 10,000 spectra. The measurement conditions are identical to the example above. The full range of the peak shift of the silicon Raman line is ± 1.5 1/cm. From these values, a residual stress of up to 0.6 GPa in the SiGe layer can be calculated.

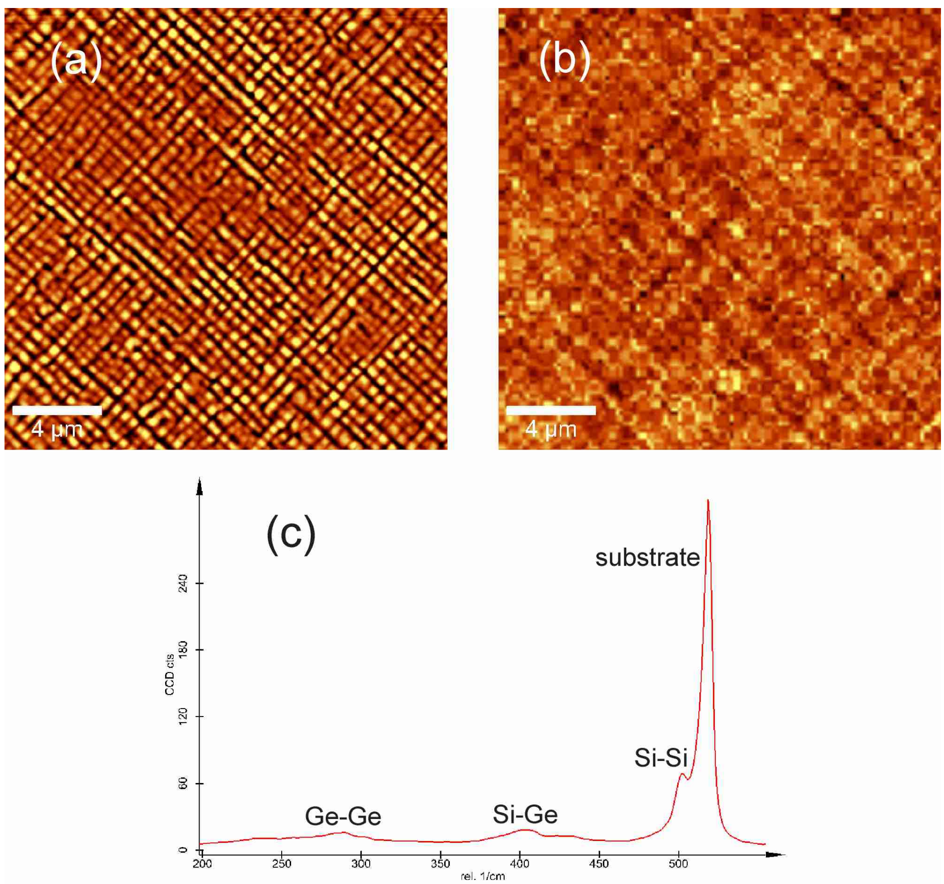

Fig. 8: (a) AFM image of the cross hatch pattern of a Si/SiGe strained epitaxial film on silicon.
Scan range is 20 µm x 20 µm and the topography scale is 30nm.
(b) Raman shift of the Si-Si stretching mode of the SiGe layer at 502 1/cm. Scan range is 20 µm x 20 µm; all other measurement parameters are as in Figure 3. Image scale is from 500.5 1/cm to 503.5 1/cm.
(c) Single Raman spectrum. The three peaks corresponding to the SiGe layer: the Ge-Ge (200 1/cm to 300 1/cm), Si-Ge (380 1/cm to 450 1/cm) and Si-Si (502 1/cm) stretching modes are indicated, along with the Si-Si (520 1/cm) peak of the substrate.