Characterization of Uni-axially Stressed Si and Ge Concentration in $Si_{1-x}Ge_x$ using Polychromator-based Multi-wavelength Raman Spectroscopy

Woo Sik Yoo, Takeshi Ueda and Kitaek Kang

WaferMasters, Inc.
246 East Gish Road, San Jose, CA 95112, U.S.A.
Phone: +1-408-451-0856 E-mail: woosik.yoo@wafermasters.com

1. Introduction

The enhanced electron and hole mobility of strained Si and strained SiGe makes them very attractive as candidate materials for high speed CMOS and HBT devices. [1-3] The amount of strain and the enhancement of carrier transport properties are tailored by the Ge content. Electrical properties can only be measured after the completion of device fabrication. It is important to establish in-line, non-destructive strain and Ge content monitoring techniques for process optimization and quality control.

Since the 1970's, Raman spectroscopy has been used in characterizing semiconductors in terms of carrier concentration, impurity content, composition, crystal structure, crystal orientation, temperature and mechanical strain. [4-5] Measurement accuracy, repeatability, spectral resolution, spatial resolution, and calibration related issues have been preventing practical application of this technique from achieving wide acceptance in the industry. Recently, Raman spectroscopy has been revisited by many researchers as a preferred, non-destructive stress/strain characterization technique for semiconductor materials. [4-13]

In this paper, we have designed a special polychromator-based multi-wavelength Raman spectroscopy system (MRS-300) to overcome the common issues with conventional Raman measurement systems. Furthermore, we have demonstrated non-destructive characterization of the lattice stress/strain in mechanically stressed Si and Ge content in SiGe in a highly accurate and repeatable manner. The application of the MRS-300 as an in-line process monitoring tool was also examined by collecting data on long term measurement stability and repeatability. An example of an in-line application of the MRS-300 system is also given.

2. Experiment

Uni-axially Stressed Si(100) Samples

Silicon coupons cut from a double side, polished 300mm Si (100) wafer (~775µm thick) were uni-axially stressed along the [110] direction. Both tensile and compressive stresses, up to 200MPa, were applied in 50MPa increments before measurement.

$Si_{1-x}Ge_x$ (100) Samples

Four $Si_{1-x}Ge_x$ (100) samples with different Ge content, ranging from 15~30 atomic percent in 5 atomic percent increments, were prepared and characterized. As a measurement reference, a stress-free Si (100) wafer was used.

Multi-wavelength Raman Spectroscopy (MRS-300)

We have designed the MRS-300 system as an in-line stress/strain monitoring system. The system has three thermoelectrically cooled, charge coupled device (CCD) cameras that can measure Raman peaks from three different excitation wavelengths without any disruption (ie, without scanning the monochromator or switching the excitation laser) (Fig. 1). The measurement capability of the system is summarized in Fig. 2. Three major spectral lines (457.9, 488.0 and 514.5nm) from a multi-wavelength Ar^+ ion laser are used as the excitation source. By selecting the wavelength of the excitation laser, the crystal quality, stress and strain in the depth direction can be characterized. [5-9]

Microscopic Raman scattering is a powerful, non-destructive technique for characterizing crystallinity and the degree of stress/strain of the crystal. [3-9] For this purpose intensity, shift, and full width at half maximum (FWHM) of Raman signals are measured.

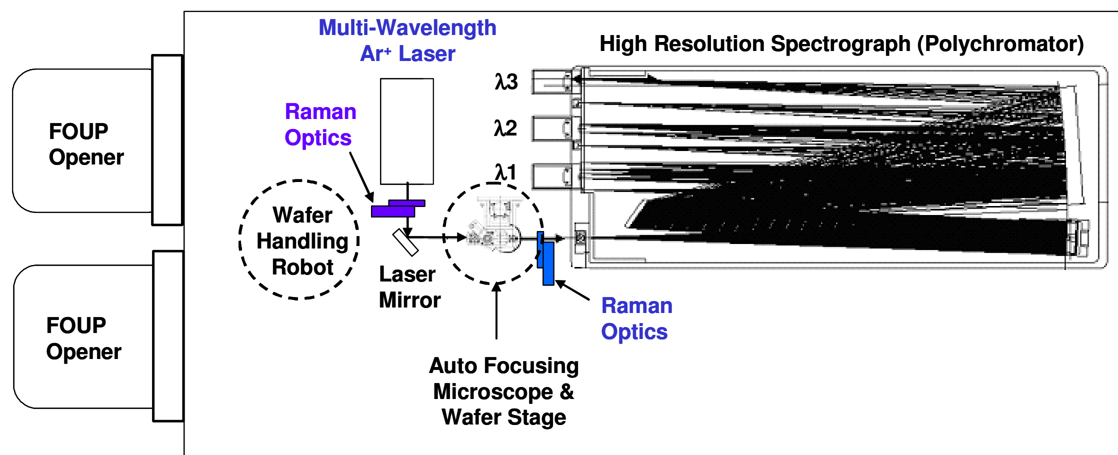

Fig. 1. Schematic illustration of multi-wavelength Raman spectroscopy (MRS-300) system.

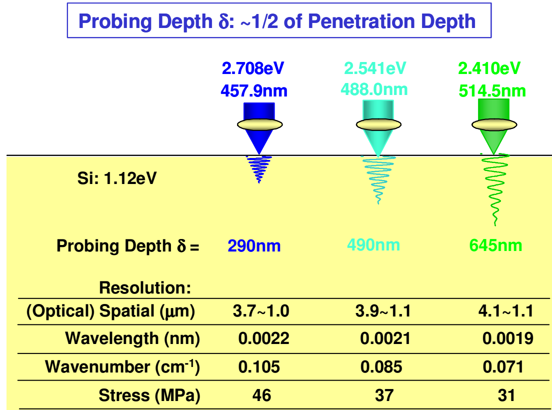

Fig. 2. Summary of measurement capability of the MRS-300 with various excitation wavelengths of the multi-wavelength Ar^+ laser.

3. Raman Results and Discussions

Uni-axially Stressed Si(100) Samples

Figure 3 shows the Raman spectra measurement configuration used in applying uni-axial mechanical stress to Si (100), along the [110] direction, using an adjustable mechanical clamping device. Raman signals from Si (100) samples under 514.5nm excitation with various uni-axial stresses (from -200MPa to 200MPa) are plotted on top of each other in the wavenumber range of 517.0~524.0cm^{-1}. As seen in Fig. 4, intensity, frequency and FWHM of Raman signals vary as the direction and magnitude of applied stress are modified.

The intensity, frequency and FWHM of Raman signals were plotted in Fig. 5 as a function of uni-axial stress applied to Si (100) along the [110] direction. The intensity of the Raman signal significantly weakened when a large tensile stress was applied causing the sample surface to become convex. Stress-free Si (100) showed a Raman peak at ~520.3cm^{-1} as reported in many studies. [4-11] Raman frequency shifted towards the higher wavenumbers as compressive stress is increased. When tensile stress is applied, the Raman frequency shifted in the opposite direction (towards the lower wavenumbers). The slope of Raman frequency versus applied mechanical stress was found to be ~430MPa/cm^{-1} and was relatively linear in the mechanical stress range of -200MPa~200MPa. The slope (or the conversion coefficient of the Raman frequency change to mechanical stress) is very close to previously reported values (within measurement error). [4, 6-8] The FWHM showed a trend of monotonic increase from 2.68cm^{-1} to 2.75cm^{-1} under mechanical stress ranging from -200MPa to 200MPa.

A good correlation between Raman frequency shift and uni-axial mechanical stress was experimentally confirmed. In reality, stress or strain in Si can be very complex. It can have a three dimensional distribution and variations. However, the average Raman frequency shift and broadening of the FWHM can be used as an indicator of stress and/or strain within the crystalline lattice. Distribution of stress and/or strain in the depth direction can be estimated by measuring Raman signals under various excitation wavelengths with different penetration depths in target materials.

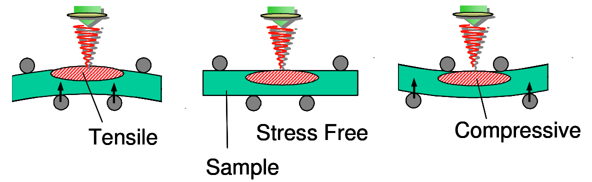

Fig. 3. Schematic illustration of Raman measurement configurations for studying the effect of uni-axial stress of Si (100) on Raman shift and FWHM.

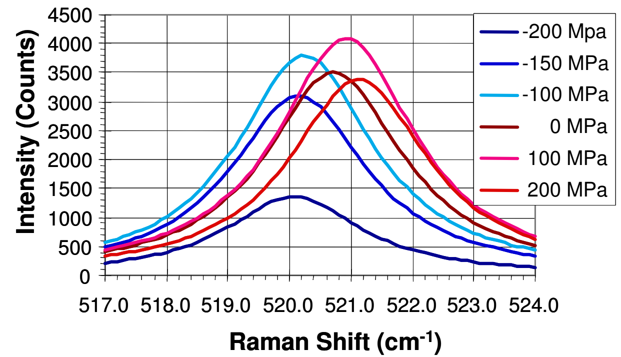

Fig. 4. Raman signals from Si (100) samples with various uni-axial mechanical stress ranging from -200MPa to 200MPa under 514.5nm excitation.

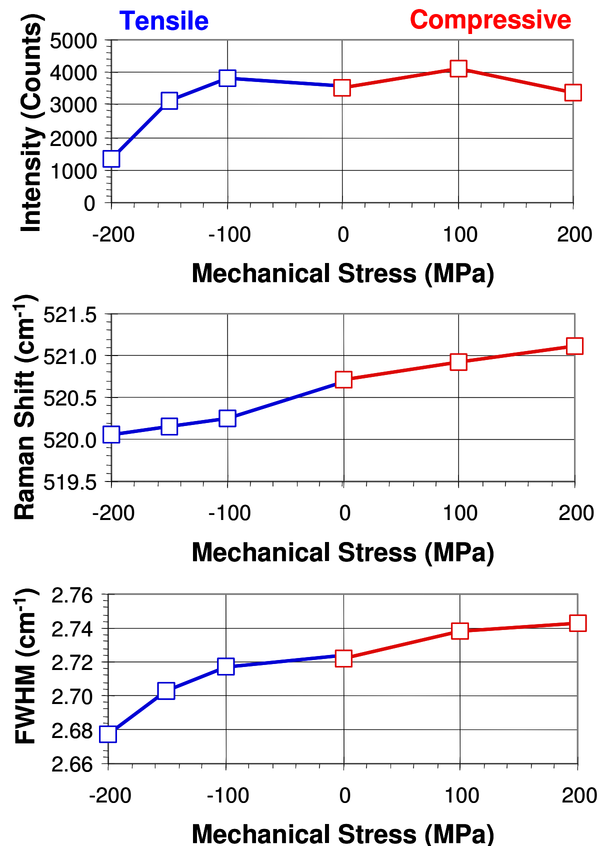

Fig. 5. Uni-axial mechanical stress dependence of intensity, frequency and FWHM of Raman signals from Si (100) samples in the range of -200MPa ~ 200MPa under 514.5nm excitation.

$Si_{1-x}Ge_x$ (100) Samples

Carrier mobility enhancement in strained Si, as well as in strained $Si_{1-x}Ge_x$, strongly depends on the amount of strain in the channel region. The amount of strain is largely affected by Ge content below, or in, the channel region. Non-destructive, accurate measurement of strain and/or Ge content is extremely important. Raman spectroscopy has been proposed as a promising characterization technique by many research groups. [1, 3, 10, 13]

In addition to a pure Si (100) sample, four $Si_{1-x}Ge_x$ (100) samples with different Ge content, ranging from 15~30 atomic percent in 5 atomic percent increments, were prepared. Raman signals under 457.9nm, 488.0nm and 514.5nm excitation were measured using the MRS-300 system. As seen in Fig. 6, all four $Si_{1-x}Ge_x$ (100) samples with different Ge content showed the second peak on the left (the lower wavenumber side) of the Raman peak from bulk Si, regardless of excitation wavelength. The second peak is identified as a Raman signal from Si-Si bonds in $Si_{1-x}Ge_x$. As the Ge content increases, the shift of the second peak from the Si-Si bonds in $Si_{1-x}Ge_x$ towards the lower wavenumber side is well known. The separation of the second peak of $Si_{1-x}Ge_x$ from the bulk Si peak is often used to determine the amount of strain and/or Ge content in $Si_{1-x}Ge_x$. Since the wavenumber resolution improves with the increase of excitation wavelength (see Fig. 1), the separation of peaks becomes greater at longer excitation wavelengths.

In our study, a monotonic increase of the separation of the second peak of $Si_{1-x}Ge_x$ from the bulk Si peak occurs at a rate of $-0.34cm^{-1}$/Ge atm% at all three excitation wavelengths (Fig. 7). Non-destructive characterization of stress and/or Ge content can easily be done in line by Raman spectroscopy between process steps and the data (both within wafer and wafer-to-wafer) can be used as a process quality assurance metric or as a feedback signal for process development and optimization. Multi-wavelength Raman measurement also enables the detection of strain or Ge content variation in the depth direction.

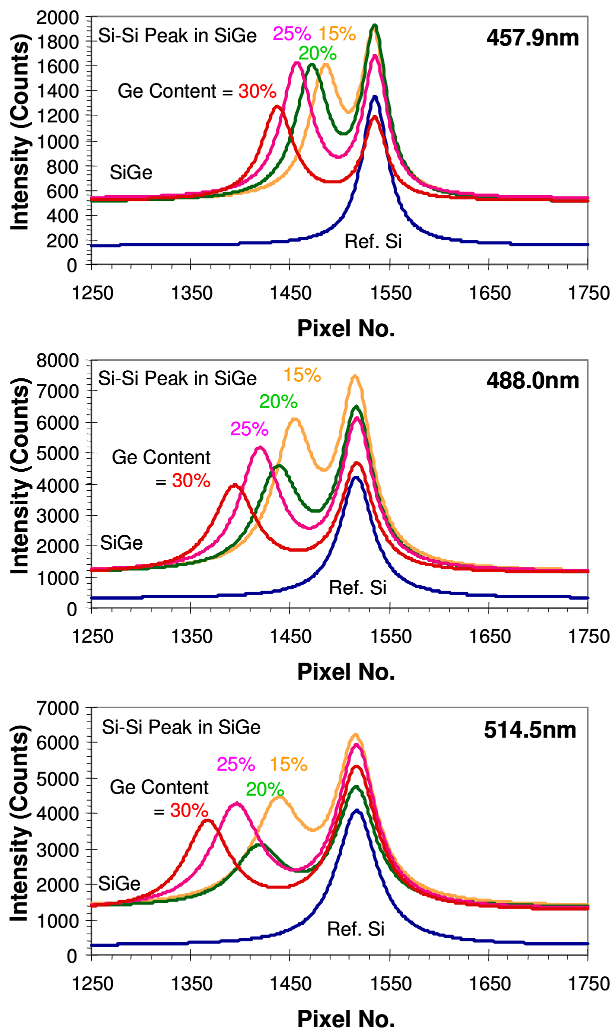

Fig. 6. Si and Si-Si Raman peaks from $Si_{1-x}Ge_x$ (x = 0, 0.15, 0.20, 0.25 and 0.30) samples under 457.9nm, 488.0nm and 514.5nm excitation. (Note: Pixel number of the CCD camera was used as horizontal axes to show the excitation wavelength dependence of spectral resolution.)

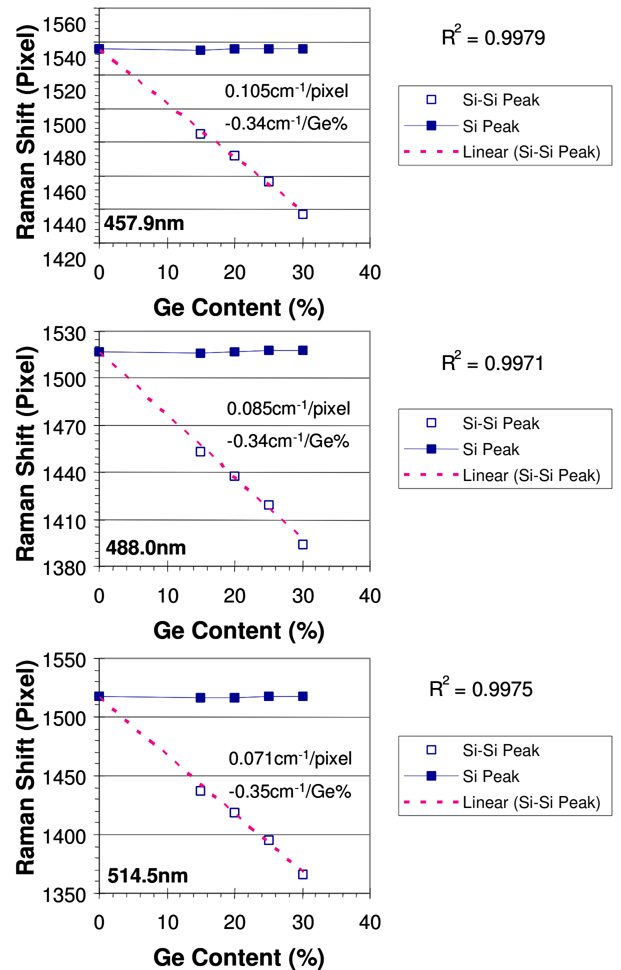

Fig. 7. Frequency shift of Si and Si-Si Raman peaks as a function of Ge content measured from $Si_{1-x}Ge_x$ (x = 0, 0.15, 0.20, 0.25 and 0.30) samples under 457.9nm, 488.0nm and 514.5nm excitation. (Note: Pixel number. of the CCD camera was used as vertical axes to show the excitation wavelength dependence of spectral resolution.)

Measurement Resolution, Accuracy and Repeatability

In any scientific experiment, establishing measurement accuracy and repeatability is essential. It is even more critical in quality control. However, the system requirements of measurement resolution, accuracy and repeatability often conflicts with the desire for of flexibility, productivity, compactness, economic factor and so on. Sometimes, system designers are forced to compromise due to economic factors and competitive decisions of equipment designers rather than scientific or engineering limitations.

In the design phase of the MRS-300 system, we have focused in maximizing measurement resolution, accuracy, repeatability, excitation wavelength flexibility and productivity. After careful review of possible design options and technical merits/demerits of individual design options, the polychromator-based multi-wavelength Raman spectroscopy system design was chosen. The unique features of a very large, long focal length focusing mirror with a wide spectrum coverage, multiple CCD cameras for measuring Raman signals from individual excitation wavelength, elimination of moving parts in the spectrograph made the realization of high spectral resolution, accuracy, repeatability, excitation wavelength flexibility and productivity possible. System calibrations required in conventional monochromator-based Raman systems (daily and between excitation wavelength switching) are eliminated. As a result, the uncertainties in absolute wavenumber of Raman signals due to calibration errors, optical misalignment due to vibration and backlash of moving parts (mainly diffraction grating(s)) are eliminated. The MRS-300 system design enabled the multi-wavelength Raman measurement from the same measurement site without refocusing or calibration. This innovative design aspect of the MRS-300 upgraded the reliability of Raman measurement and opened the in-line monitoring of properties of Si (stress, strain, Ge content etc.) for process development, process optimization and quality control.

Raw data from Raman signals from bare Si (100) measured using the MRS-300 system under various wavelength excitations is shown in Fig. 8. Raman signals from all excitation wavelengths are centered at ~520.3cm^{-1} and are highly symmetrical suggesting that the MRS-300 system has extremely low distortion error and that the quality of the reference Si (100) is excellent. Since the pixel size of all CCD cameras are the same (13.5µm x 13.5µm), data points are much denser in longer wavelength Raman data after converting the same pixel bandwidth into wavenumbers or Raman frequency. The wavenumber resolution is better than 0.105cm^{-1} in all three wavelengths (457.9nm, 488.0nm and 514.5nm) before curve fitting. Since the 0.1cm^{-1} shift of the Raman signal from Si corresponds to ~40MPa, high wavenumber resolution and high measurement repeatability of at least ~0.1cm^{-1} before curve fitting is highly recommended for accurate stress and strain analysis of Si related devices.

For measurement repeatability verification of the MRS-300 system, Raman signals from the same reference Si (100) sample were measured under various wavelength excitations daily for a period of one month. Figure 9 summarizes the results. Both variations in Raman shift and FWHM at all wavelengths in the monitoring period (one month) was less than 0.05cm^{-1}. Superior performance of the MRS-300 system design was experimentally verified. With the high spectral resolution, measurement accuracy and repeatability of MRS-300 system, reliable Raman study on stress, strain and the effect of Ge is possible. In-line monitoring of process variations within wafer, wafer-to-wafer and system-to-system can provide additional insights into hidden material, process and equipment problems.

Figure 10 shows an in-line monitoring example along device fabrication process steps (514.5nm excitation). Raman signals were measured on a virgin 300mm Si (100) wafer. As process steps progress, the average and range of both Raman shift and FWHM varied significantly. Process steps, process equipment and specific wafers with potential problems can be identified by monitoring the change in the average and range of the Raman shift and FWHM along process steps before the completion of device fabrication and electrical performance tests of devices.

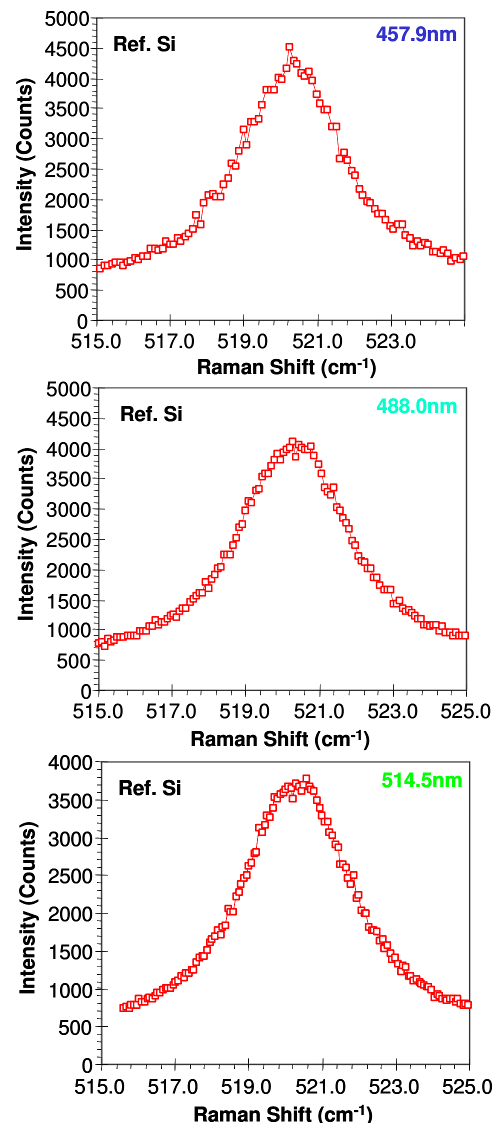

Fig. 8. Raw Raman measurement data taken using MRS-300 system under 457.9nm, 488.0nm and 514.5nm excitation demonstrating very high wavenumber resolution.

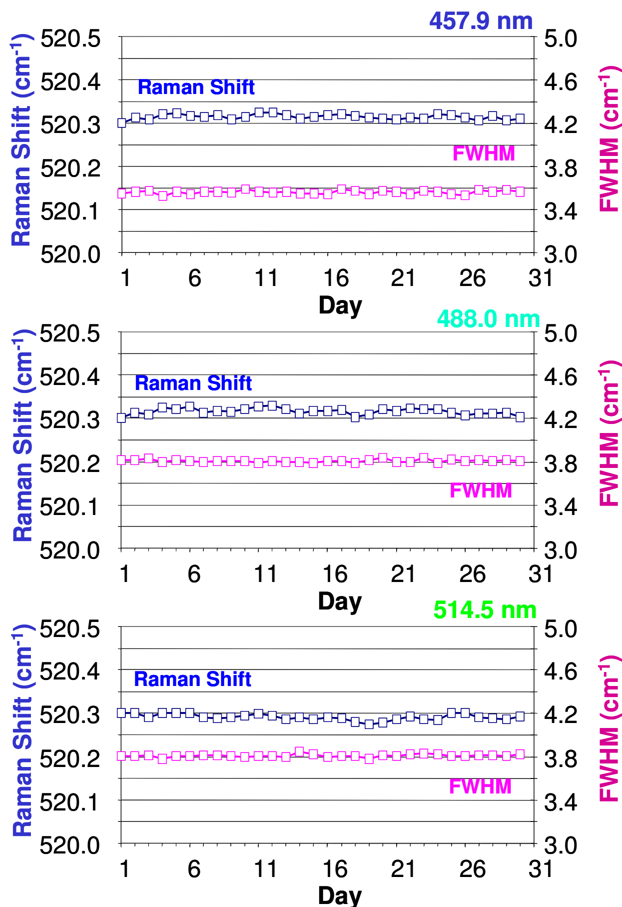

Fig. 9. Measurement repeatability data collected from MRS-300 system for a period of one month.

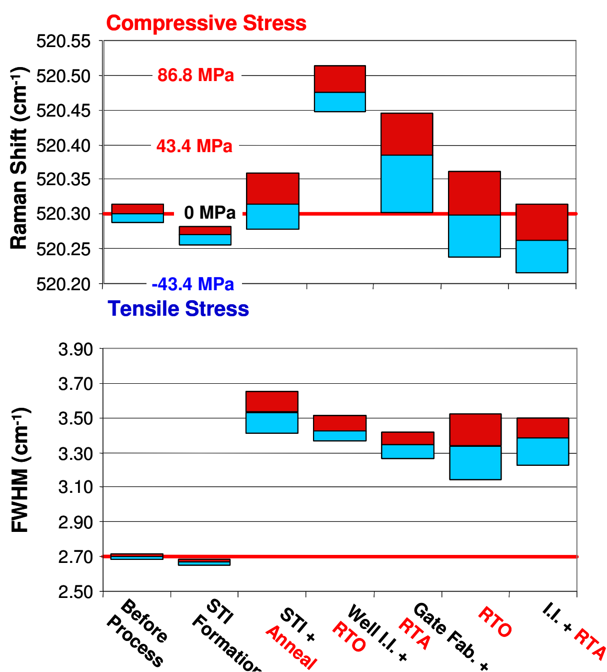

Fig. 10. In-line monitoring example along device fabrication process steps. (514.5nm excitation)

4. Summary

The design concept of a polychromator-based multi-wavelength Raman spectroscopy system (MRS-300) which is designed to overcome the common issues with conventional Raman measurement systems is described. The system is specially designed for non-destructive, material and process characterization applications in the semiconductor industry. The performance of the MRS-300 system and non-destructive nature of the tests of lattice stress/strain and Ge content were demonstrated using mechanically stressed Si samples and epitaxially grown $Si_{1-x}Ge_x$ samples. The capability of very high measurement resolution (0.105cm^{-1}/pixel or better), accuracy repeatability (0.05cm^{-1} or better) and repeatability (0.05cm^{-1} or better) of the system were demonstrated at all three excitation wavelengths (457.9nm, 488.0nm and 514.5nm). Possibility of MRS-300 applications as an in-line process monitoring system was also introduced with an example of long term measurement stability and repeatability data.

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