Non-destructive evaluation methods for subsurface damage in silicon wafers: a literature review

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Abstract: The Subsurface Damage (SSD) in silicon wafers induced by any mechanical material-removal processes has to be removed by subsequent processes. Therefore, the measurement of SSD is critically important for cost-effective manufacturing of silicon wafers. This review paper presents several Non-Destructive Evaluation (NDE) methods for SSD in silicon wafers, including X-ray diffraction, micro-Raman spectroscopy, photoluminescence and laser scattering.

Keywords: manufacturing; measurement; non-destructive method; silicon wafer; subsurface damage; SSD.

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1 Introduction

As the primary substrates for Integrated Circuits (ICs), silicon wafers are manufactured through a series of processes, such as crystal growth, slicing, flattening (grinding/lapping), etching and polishing. Subsurface Damage (SSD) up to tens of microns deep induced during mechanical material-removal processes (such as slicing, grinding and lapping) must be removed by subsequent processes. For IC manufacturing, the completed device wafers are usually thinned by a back-grinding process before separated into individual chips/dies. The SSD induced by back-grinding must be kept below a certain level to ensure the wafers or chip/dies possess sufficient strength. Therefore, there is a critical need to assess SSD induced by these mechanical material-removal processes.

Many techniques have been reported to observe/measure SSD in silicon wafers: angle polishing (Abe et al., 1994; Tonshoff et al., 1997), angle lapping (Jeong et al., 2000; Oh et al., 1999), step polishing (Stephens, 1986), step etching plus scanning infrared depolarisation (Lundt et al., 1994), cross-sectional microscopy (Abe et al., 1994; Kang et al., 2005; Mchedlidze et al., 1995; Pei et al., 1999; Zarudi and Zhang, 1996) and etching method (Tonshoff et al., 1990). However, these techniques either destroy the wafers or measure only localised areas instead of the whole wafer.

In order to enhance the cost-effectiveness in the manufacturing of semiconductor wafers and ICs, it is desirable to have Non-Destructive Evaluation (NDE) methods to characterise subsurface cracks induced by mechanical material-removal processes.

This review paper presents the basic principles and applications of four NDE methods for silicon wafers (X-ray diffraction, micro-Raman spectroscopy, photoluminescence and laser scattering) in Sections 2–5, respectively. It also discusses the advantages and disadvantages of these four methods in Section 6.

2 X-ray diffraction

2.1 Introduction of X-ray diffraction

As mentioned in Section 1, some manufacturing processes (such as slicing, grinding and lapping) will induce SSD in silicon wafers, involving microstructure change, dislocations and microcracks. Some causes for SSD are mechanical stresses developed during and after processing (Hu, 1991).

X-ray diffraction is a non-destructive method to determine residual stress from the lattice deformation of a crystal. A crystal lattice is a regular three-dimensional distribution (cubic, rhombic, etc.) of atoms in space. When X-rays come in at a particular angle, they are reflected specularly (mirror-like) from the different planes of crystal
atoms. However, for a particular set of planes, the reflected waves interfere with each other. A reflected X-ray signal is only observed if Bragg’s condition is met for constructive interference.

Figure 1 illustrates how X-ray diffraction works. Figure 1(a) shows X-rays incident upon a simple crystal structure; Bragg’s condition is met for both ray A and ray B. Figure 1(b) shows the diffraction geometry. The extra distance travelled by ray B must be an exact multiple of the wavelength of the radiation. This means that the peaks of both waves are aligned with each other. Bragg’s condition can be described by Bragg’s Law: 

\[ 2d \sin \theta = m\lambda, \]

where \( d \) is the distance between planes, \( \theta \) is the angle between the plane and the incident (and reflected) X-rays, \( m \) is an integer called the order of diffraction and \( \lambda \) is the wavelength.

Figure 1
Illustration of X-ray diffraction (a) X-rays A and B incident upon a crystal and (b) diffraction geometry

Figure 2(a) shows a standard X-ray diffraction pattern with aligned sharp peaks for a perfect crystal structure. During some manufacturing processes, residual stress may be resulted from non-uniform, permanent three-dimensional changes in silicon wafers. These changes usually occur as plastic deformation and may also be caused by cracking and local elastic expansion or contraction of the crystal lattice (Bismayer et al., 1994). In these cases, the crystal structure is no longer perfect (\( d \) is changed) and the diffraction peaks are broadened and shifted, as shown in Figure 2(b). Thus, by examining the changes of the X-ray diffraction pattern, the residual stress can be characterised. Further, the related defects can possibly be identified.

Figure 2
Illustration of X-ray diffraction patterns: (a) for a perfect crystal and (b) for an imperfect crystal
2.2 Application of X-ray diffraction

A high-resolution X-ray diffraction system (Tonshoff et al., 1990) (shown in Figure 3) was used to detect the damaged layer of silicon wafers. The X-ray diffraction pattern of ground silicon wafers showed an increased diffraction intensity in ‘the wings of the Bragg-peak’ caused by the damaged layer. Comparing with that of the ground wafer, the diffraction pattern of a polished ‘perfect’ wafer showed a considerably smaller width at half maximum of the Bragg-peak (as shown in Figure 4). Bismayer et al. (1994) reported that the quantitative determination of residual stresses in silicon wafers could be detected by the X-ray diffraction technique. For silicon wafers, the penetration depth by X-ray diffraction lies between 30 and 60 \( \mu \text{m} \) (Bismayer et al., 1994).

Figure 3  Schematic diagram of a high-resolution X-ray diffraction system

Source: Tonshoff et al. (1990).

Figure 4  X-ray diffraction pattern of silicon wafers

Source: Bismayer et al. (1994).
Mikulík et al. (2002) demonstrated the capability of high-resolution X-ray diffraction techniques with synchrotron radiation sources to inspect the structural perfection in semiconductor wafers. They applied X-ray diffraction to visualise and characterise the defects (dislocations and microcracks) in semiconductor wafers (in particular, silicon and GaAs wafers) induced by growing, cutting and grinding. The number and the sharpness of observed Pendellösung fringes depend on the crystal perfection. Figure 5 shows the SSD characterisation of silicon wafers by Pendellösung fringes. The visibility of fringes is the best for the ‘perfect’ monitor wafer. No fringes are visible on the ground wafer. Etching-off or polishing-off (by CMP) only a surface layer of 5 μm in thickness after grinding is sufficient to remove the SSD significantly, resulting in a partial restoring of Pendellösung fringes.

Figure 5 SSD characterisation by Pendellösung fringes: (a) ‘perfect’ monitor wafer (20 fringes); (b) ground wafer (no fringe) and (c) ground and 46 μm etched wafer (7 fringes)

Source: Mikulík et al. (2002).

X-ray diffraction has been used to study silicon-on-insulator structures by Popov et al. (2001) and to study silicon carbide on insulator structures by Milita et al. (2002). Their experimental results showed that X-ray diffraction could characterise the lattice defects in these wafers and quantify these lattice defects and related stresses at the bonding interface.

3 Micro-Raman spectroscopy

3.1 Introduction of Raman spectroscopy

The principle of Raman effect is based on an inelastic light-scattering process. When light is scattered from a molecule, most light (photons) is elastically scattered. That is, most of the scattered light has the same frequency and wavelength as the incident light. However, a small fraction of light is scattered at optical frequencies different from and usually lower than, the frequency of the incident light. This inelastic scattering of light is called the Raman effect. Further, the microscopic changes (long- and short-range
disorders), impurities and residual strains strongly influence the Raman spectrum of a material. These microscope changes lead to changes in phonon frequencies, broadening of Raman peaks and breakdown of Raman selection rules. When a beam of monochromatic light passes through a crystal, the Raman effect occurs. Thus, the presence of amorphous silicon (a-Si) as well as residual stresses can be detected by this technique (Yan, 2004).

The basic experimental set-up for a micro-Raman spectroscopy system is shown in Figure 6. The laser light is focused on the sample through a microscope (often a confocal one where a spatial pinhole is used to eliminate the out-of-focus light thus increasing the contrast), resulting in a spot size down to about 1 μm (spot sizes as small as 0.3 μm can be obtained if an oil immersion objective is used). Generally, the spatial resolution of micro-Raman can be 1 μm (Wolf, 1996). The scattered light of the sample is collected, through the same microscope focused on the entrance slit of a spectrometer (detection device). The sample can be mounted on a computer-controlled X–Y stage, which allows it to be scanned in small steps (typical 0.1 μm) in a given direction. The total collection volume depends on the light-scattering properties of the material being analysed (Harris, 2004). Furthermore, the light penetration depth changes with different wavelengths. Thus, it is possible to probe different depths of the sample by varying the wavelength (Sparks and Paesler, 1988, 1993).

Figure 6 Illustration of micro-Raman spectroscopy

Source: Bismayer et al. (1994).
3.2 Application of micro-Raman spectroscopy

Figure 7 shows a typical Raman spectrum obtained in back scattering from a (100) silicon wafer using the 475.8 nm line of an argon laser. The silicon Raman peak is very strong and has a frequency of about $\omega_0 = 520 \text{ Rcm}^{-1}$ in the absence of stress (The unit ‘Rcm$^{-1}$’ denotes ‘relative cm$^{-1}$’, since the frequency is always measured relative to the frequency of the laser light) (Yan, 2004). This typical Raman spectrum from a (100) silicon wafer can be used as the reference when studying the Raman spectrums from silicon wafers with defects.

Figure 7  Typical Raman spectrum from a (100) silicon wafer


Bismayer et al. (1994) used micro-Raman spectroscopy to measure the silicon wafers machined by ID sawing, grinding and polishing (the polished wafer was used as the reference). Comparing with the polished wafer, different Raman peak position and intensity were observed for the ground wafer (shown in Figure 8). This Raman peak shift was caused by the SSDs induced by high stresses left in the wafer during the machining process. The damage depth could be obtained using depth-profiling techniques outlined by Sparks and Paesler (1993). The phase transformations in silicon within the penetration depth could also be analysed by using micro-Raman microscopy. The penetration depth changes with the laser beam wavelength, which allows data acquisition from depths of <0.1 to 10 $\mu$m (Gogotsi et al., 1999).

Comparing with the typical micro-Raman spectrum shown in Figure 7, Yan (2004) estimated whether the subsurface layer with the laser penetration depth in the single-point diamond machined silicon substrates was transformed into an amorphous state or not. For example, in Figure 9, a micro-Raman spectrum for a layer of 50 nm thick on a silicon wafer exhibited another significant broadband peak centred at 470 cm$^{-1}$ (besides the typical peak at 520 cm$^{-1}$ for perfect silicon), indicating that the subsurface layer was partially transformed into an amorphous state.
4 Photoluminescence

4.1 Introduction of photoluminescence

The optical absorption processes in semiconductors result in the excitation of an electron from a lower-energy state to a higher-energy state (from the valence band to the conduction band). This mechanism is attributed to free carriers that are able to make transitions to higher energy levels through the absorption of a photon with subband-gap energy (near-infrared and longer for silicon) (Shaughnessy et al., 2006). Then the photon
is radiated back out. During the period between absorption and emission, luminescence is emitted and could be detected. The photoluminescence spectra can be recorded, just like X-ray diffraction patterns and micro-Raman spectrum.

Early photoluminescence utilizations were done under cryogenic temperature (such as, at liquid helium temperature), which made its application in semiconductors rather limited. Recently, photoluminescence measurements were performed using a Silicon Photoenhanced Recombination (SiPHER) instrument (Figure 10), operating at room temperature. In this instrument, the excitation laser beam was focused onto sample surface through a microscopic objective to create a small excitation volume (1–2 μm in diameter). Then the sample was scanned in the x–y direction. And the photo-generated carriers could remain within the probing volume, for a differential optoacoustic modulator controlled the laser source, the carriers were strongly confined and the carrier diffusion length was greatly reduced, then carrier recombination (a process that both carriers, electrons and holes, annihilate each other) was enhanced. Next the emitted luminescence was collected by the same lens and measured using a sensitive rapid detection system. Sampling the luminescence from only a small region within the overall diffusion envelope further enhanced the spatial resolution. Because of the localised degradation in carrier lifetime, defects were detected and observed as darkened regions at the physical position. A high-resolution image was obtained, and enhanced by digital filtering. The system was able to perform whole-wafer maps or high-resolution (1 μm) scans. A Charge-Coupled Device (CCD) camera and white light source were incorporated to allow imaging of the wafer surface and enable separation of the photoluminescence – generated singles from the surface defects (Nevin et al., 2003).

Figure 10  Schematic diagram of the photoluminescence measurement system

Source: Nevin et al. (2003).
4.2 Applications of photoluminescence

The photoluminescence technique has been employed for defect identification and non-contact characterisation of silicon materials, including the determination of boron, phosphorus and carbon after irradiation, the analysis of metal-decorated stacking faults and misfit dislocations (Kirscht et al., 2002).

Ostapenko et al. (2000) used photoluminescence to monitor defects in polycrystalline silicon wafers. They found that the photoluminescence spectrum at room temperature ($T = 290\,\text{K}$) was generally composed of two bands in as-grown and processed polycrystalline silicon wafers, as shown in Figure 11. The high-energy maximum at $1.09\,\text{eV}$ (curve 1) corresponds to the well-known band-to-band emission caused by phonon-assisted recombination of bound and unbound free electrons and free holes, which is a typical photoluminescence spectrum in a high-lifetime region of polycrystalline silicon. A second broad band at lower energies (curve 2), with the maximum at about $0.8\,\text{eV}$, is referred to as the ‘defect’ band. The $0.8\,\text{eV}$ band is strongly localised in low-lifetime regions. So the photoluminescence defect band can be used for identifying low-lifetime regions of polycrystalline silicon (Ostapenko et al., 2000).

Figure 11 Room-temperature photoluminescence (PL) spectra in the area with high (1) and low (2) minority carrier lifetime

Buczkowski et al. (2003) modelled a photoluminescence spectrum of silicon wafers under conditions of steady-state and depth-dependent excitation, while ignoring surface recombination and carrier diffusion. They found that experimental data corresponded with simulated data well, in spite of model limitations. The data showed that the photoluminescence spectrum reached a maximum within $0.01–0.1\,\Omega\,\text{cm}$ resistivity range. At the lower resistivity, the photoluminescence spectrum primarily depended on
doping level and it became very useful for analysing doping uniformity and striations. At the high resistivity, the photoluminescence spectrum depended on the applied excitation levels and the concentration of deep recombination levels (material contamination) and it could be used for defect and contamination analysis. Within the whole range of resistivity, photoluminescence spectrum could be used for imaging of extended defects, such as dislocations and precipitates (Buczkowski et al., 2003).

The photoluminescence technique was also applied for interfacial defects in silicon-on-silicon fusion-bonded silicon wafers. Nevin et al. (2003) showed that threading dislocations, slip dislocations and oxide agglomerates in 40 μm thick silicon layer could be detected and mapped with high resolution by photoluminescence methods.

Kirsch et al. (2002) reported their successful application of photoluminescence to detect oxygen precipitation related defects, stress relaxation related defects and doping striations in various silicon materials, including lightly doped, precipitation-annealed polished wafers intentionally contaminated with Fe, Ni and Cu and several types of epi wafers based on heavily doped substrates. Figure 12 is an example of stress relaxation related defect detected by the photoluminescence micrograph. Stress relaxation related epi misfit dislocation networks are revealed with a spatial resolution close to 1 μm. In the arsenic substrate example (Figure 12(b)), even single dislocations are visible. Dislocations in the antimony substrate example (Figure 12(a)) seem to be heavily decorated, based on the strong photoluminescence contrast (Kirscht et al., 2002).

![Figure 12](image-url)

**Figure 12** Photoluminescence micrographs of depth profiling of processed device wafers: dislocations in (a) antimony substrate and (b) arsenic substrate

Source: Kirscht et al. (2002).

## 5 Laser scattering

### 5.1 Introduction of laser scattering

With laser scattering techniques, when a laser beam directly illuminates a sample, the amount and distribution of back-scattered light could be monitored. The basic principle of light-scattering technology is that different microstructures in a test sample
may cause a change in the scattering characteristic of the reflected light. Especially, a sudden change in the light intensity would occur when the incident light encounters a defect (Wang et al., 2000).

Figure 13 illustrates a laser scattering system. A laser beam passes through a pinhole and illuminates a small area of the sample. Light is reflected from the illuminated spot on the sample to the objective, where it is directed by a beamsplitter toward the confocal pinhole aperture. The pinhole positioned in front of the detector gives the system its confocal property by rejecting light originating from neighbouring focal planes. Light rays from an unfocused plane are blocked from reaching the detector. However, all light rays originating from the focal plane pass through the pinhole aperture and are collected by the detector. The ability to closely discriminate between the light rays originating at the focal plane from those originating from the unfocused planes enables a laser scattering system to be a powerful tool in generating depth-precise images (Stove, 1990). Usually, objective lenses with a high Numerical Aperture (NA) are used to provide good resolution in the $x$-, $y$- and $z$-directions.

**Figure 13** Schematic diagram of a laser scattering system

*Source: Stove (1990).*
Laser scattering techniques require that the sample material should be translucent. Zhang et al. (2002) found that silicon wafers have the appropriate optical transmission properties for laser scattering. The relative depth of a subsurface defect can be approximated by determining the ratio of the scatter intensity in the region of the defect to that of the normal material. Thus, by measuring the type, degree, size and shape of a defect’s laser scatter signature, a general description of the defect can be derived, along with its location in the sample (Stove, 1990).

5.2 Applications of laser scattering

The laser scattering method has been used by Goto et al. (2001) to measure subsurface microdefects in Czochralski-grown (CZ) silicon wafers and epitaxial silicon wafers. Utilising the temperature dependence of the absorption coefficient of silicon at the laser wavelength, they could measure the depths and dimensions of microdefects in the region to a depth of 5 μm were measured. They found that the minimum size of detectable microdefects at a penetration depth \(d\) exponentially increased with \(d\) in CZ and epitaxial silicon wafers. Figure 14 shows a result of the number of the defects versus the penetration depth for the CZ wafer. For epitaxial wafers, distinct boundaries between their epitaxial layers and substrates were successfully detected (Satio et al., 2002).

**Figure 14** Number of defects versus penetration depth in the CZ wafer

![Graph showing number of defects versus penetration depth in the CZ wafer](image.png)

*Source: Satio et al. (2002).*

Sun et al. (1998) have set up a two-detector laser scattering system as illustrated in Figure 15. The vertically polarised laser beam was directed through a Polarising Beam-Splitter (PBS) cube and focused on the sample surface. Unless the surface was extremely rough, the scattered/reflected light from the surface would not change its
polarisation. Therefore, all the surface-scattered light would be reflected in the PBS and
directed back towards the laser. However, any light scattered from the subsurface
material underwent several reflections and refractions at microstructural discontinuities
(such as cracks), so the subsurface scattered light became depolarised. Half of the
subsurface back-scattered light would be reflected by the PBS and directed back to
the laser, the other half would be transmitted by the PBS into the detection train. The
horizontally polarised back-scattered light that passed through the surface-illuminating
PBS entered into the second PBS (detecting PBS). Then it was directed through a
quarter-wave ($\lambda/4$) plate, imaged by a positive lens onto a polished stainless steel pinhole
aperture and recorded by Detector A. Only the light scattered from the subsurface
directly beneath the incident spot passed through the aperture and onto Detector A.
The remaining light scattered from the area around the illuminating spot was
reflected back through the lens and $\lambda/4$ plate. In this case, its polarisation was rotated to
vertical and it was reflected by the detecting PBS and directed to a 50/50 beam
splitter. One side of this splitter was imaged by a positive lens onto Detector B, while
the other side was imaged onto a CCD array to monitor the scattering pattern
(Sun et al., 1998).

**Figure 15** Illustration of a two-detector laser scattering system

Zhang and Sun (2005) used this system to measure the SSD of ground silicon wafers and
obtained promising results. A scattering image of a ground silicon wafer is shown in
Figure 16. The white speckles represent subsurface defects or cracks and the white
lines represent the scratches generated when the grinding abrasives pass through the
wafer surface. They also found that the penetrated depth of laser into silicon wafers
was increased from 9 $\mu$m to 120 $\mu$m, with increasing the laser wavelength from 630 nm
to 900 nm.
6 Conclusion

Four NDE methods are introduced in this paper: X-ray diffraction, micro-Raman spectroscopy, photoluminescence and laser scattering. They have been applied to measurement of SSD in semiconductors with encouraging results. However, each of them has its own shortcomings. Table 1 compares these four NDE methods.

Although, considerable research has been conducted to use these non-destructive methods for SSD measurement in silicon wafers, many problems remain. For example, high resolution is needed for X-ray diffraction. It is extremely time-consuming to scan the entire wafer with micro-Raman spectroscopy. Photoluminescence is not effective at room temperature. While the laser scattering can potentially be an efficient NDE method to measure the SSD for whole silicon wafers, further development is needed before it can be applied in industry.

Table 1 Comparison of four non-destructive methods

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References


