PROCESS FOR MAPPING METAL CONTAMINANT CONCENTRATION ON A SILICON WAFER SURFACE

BACKGROUND OF THE INVENTION

The process of the present invention is generally directed to the detection of metallic contaminants on a surface of a semiconductor substrate. More particularly, the present process is directed to mapping the surface of a single crystal silicon wafer in order to determine the distribution of metallic contaminants thereon. The present process is further directed to the preparation of a reference, or calibration, wafer to be utilized in the mapping process.

Semiconductor wafers suitable for the fabrication of integrated circuits are produced by slicing thin wafers from a single crystal silicon ingot. After slicing, the wafers undergo a lapping process to give them a substantially uniform thickness. The wafers are then etched to remove damage and produce a smooth surface. The next step in a conventional wafer shaping process is a polishing step to produce a highly reflective and damage-free surface on at least one face of the wafer. It is upon this polished face that electrical device fabrication takes place.

It is well known that the presence of contaminants, in particular metallic contaminants, on the surface of a semiconductor wafer can greatly diminish the quality and performance of integrated circuits fabricated therefrom. Manufacturers of integrated circuits therefore place strict limitations upon the surface concentration of metallic impurities, rejecting those wafers which exceed this concentration. As a result, silicon wafers are typically cleaned after some or all of the preparation

steps to help reduce the amount of surface contamination present on the final wafer product.

A number of methods exist for analyzing the wafer surface to determine the concentration of metallic impurities thereon. These methods may generally be divided into two groups based upon whether they provide data on the local or overall concentration of surface contaminants. For example, methods which provide data on the overall, or gross, concentration of surface contaminants typically involve the dissolution of an oxide layer present on the wafer surface by means of either hydrofluoric acid vapor or solution, the condensed vapor or solution then being analyzed to determine the concentration of metallic contaminants therein. Such methods are favorable because they typically enable the detection of much lower levels of metallic contaminants; that is, such methods are favorable because they typically have much lower detection limits relative to those methods which determine the local concentration of metallic contaminants. Additionally, such methods efficient provide data as to the contaminant concentration for the entire wafer surface.

Methods which provide data on the local concentration of metallic contaminants typically involve analyzing the wafer surface by means of secondary ion mass spectrometry, Auger spectroscopic analysis, neutron activation analysis, or total X-ray reflection fluorescence spectroscopy (TXRF) analysis. Such methods are favorable because they are essentially nondestructive to the wafer surface, which means multiple analyses can be performed on the same wafer. For example, after any step of the wafer manufacturing process, the wafer may be analyzed before proceeding to the next step. Such methods thus provide the means by which to monitor the

manufacturing process and identify potential sources of metallic contamination. In addition, because these methods provide local results for the wafer surface, the surface can effectively be "mapped"; that is, these methods may be used to determine where on the surface the metallic contaminants are located. Such data is helpful because a particular contamination pattern may suggest the source of the contaminants. For example, a circular pattern of contaminants in the center of the wafer might suggest that the wafer handling chuck is the source of the contamination, while a generally uniform distribution of contaminants might suggest the etching solution or polishing slurry is the source of the contaminants.

Accordingly, these local methods of surface analysis provide very beneficial information about not only the wafer, but also the wafer manufacturing process. However, in order for these methods to provide reliable and reproducible results, the methods and instrumentation employed must be properly calibrated. Generally speaking, the instrumentation utilized in such methods must be calibrated because of any of a number of factors which can affect the measurement such as, for example, instrument background noise, variations in detector and collection efficiency, and variations in the response from the impurity being detected. Precise calibration typically requires the preparation of a reference standard, or wafer in this case, which must meet very stringent specifications as to the concentration of metallic contaminants on its surface. In this way, the actual response of the equipment can be calibrated against the known impurity concentration of the reference wafer.

One of the important conditions placed upon these reference wafers is that they are uniformly contaminated

over the entire surface, in order to ensure an accurate and a reliable calibration regardless of the location utilized on the reference wafer surface. It is also desirable for the surface to be uniformly contaminated with all of the various metallic contaminants specifically of interest for a given wafer manufacturing facility. As a result, while reference wafers are commercially available, they do not necessarily possess the characteristics needed for a given manufacturing site, where testing is to occur. Additionally, they are not necessarily capable of allowing the calibration to be achieved with high precision.

Furthermore, gettering is often a desirable characteristic of silicon wafers, a characteristic which is investigated by various performance tests. However, reference wafers for such tests are not readily available. Therefore, results of such tests need to be cross-checked to improve the precision of such tests using means such as ICP-MS or flameless atomic absorption spectroscopy.

Accordingly, a need continues to exist for a reference or calibration wafer which can easily be prepared to have a uniformly contaminated surface, and which can be utilized as a comparative control suitably matching the manufacturing conditions and gettering characteristics of the wafers which are ultimately to be evaluated.

SUMMARY OF THE INVENTION

Among the objects of the present invention, therefore, may be noted the provision of a process for preparing a reference wafer suitable for use in the nondestructive surface analysis of a silicon wafer to determine the concentration and distribution of metallic

contaminants thereon; the provision of such a process which is economical to perform and time efficient; the provision of such a reference wafer having a uniformly contaminated surface; the provision of such a reference wafer which is particularly suitable for use in the surface analysis of a silicon wafer by means of total reflective X-ray fluorescence; and, the provision of a process for mapping the surface contaminant concentrations of a silicon wafer surface wherein such a reference wafer is employed.

Briefly, therefore, the present invention is directed to a process for preparing a reference wafer suitable in the calibration of a total reflective X-ray fluorescence spectrometer. The process comprises preparing an aqueous salt solution comprising water and a water-soluble metallic salt and then mixing a portion of the aqueous salt solution with an organic solvent to form a treatment solution. A substantially metal-free silicon wafer is immersed in the treatment solution and removed. The treated wafer is then allowed to dry.

The present invention is further directed to a process for determining the distribution of a metallic contaminant on the surface of a silicon wafer by means of a total reflective X-ray fluorescence spectrometer. The process is characterized in that a reference wafer used to calibrate the spectrometer is prepared by immersing a virgin wafer for a predetermined period of time in a mixture obtained by mixing water-soluble salts of desired metals with an organic solvent selected from a lower alcohol or a ketone, and then drying the thus treated wafer before it is used.

Other objects and features of the present invention will be in part apparent and in part pointed out hereinafter.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a diagram in the shape of a histogram illustrating the results of investigating the distribution of nickel deposits on the surface of a reference wafer prepared in accordance with the process of the present invention, as determined by total X-ray reflection fluorescence spectroscopy.

FIG. 2 is a diagram in the shape of a histogram illustrating the results of investigating the distribution of nickel deposits on the surface of a reference wafer prepared by spin coating, performed by means common in the art, as determined by total X-ray reflection fluorescence spectroscopy.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The process of the present invention enables the preparation of a reference or calibration wafer, the surface of which is substantially uniformly contaminated with one or more metallic impurities of interest, for purposes of calibrating instrumentation utilized in determining both the concentration and distribution of metallic contaminants on the surface of a silicon wafer prepared by conventional manufacturing methods. More specifically, the present process enables the preparation of a reference wafer having a substantially uniformly contaminated surface to be utilized in the calibration of total X-ray reflection fluorescence instrumentation, thus making the TXRF instrumentation suitable for use in mapping the surface of a silicon wafer for purposes of determining the concentration and distribution of metallic contaminants thereon.

In accordance with the process of the present invention, an aqueous solution is prepared using commonly

available water-soluble salts of the metals (of a purity commonly used for purposes of preparing calibration standards for this type of instrumentation) for which the sample wafers are to be analyzed. Conventional silicon wafer manufacturing conditions or environments typically results in the contamination of the wafer surface with such metals as zinc, nickel, copper, iron, calcium and chromium, among others. Therefore, this aqueous solution can be prepared by adding one or more of the respective salts to a quantity of purified water. Typically, however, stock or standard solutions of each metal salt are prepared to have a concentration of about 10 ppm (parts per million).

Once prepared, each stock solution is mixed with a quantity of a commercially available volatile organic solvent (of a purity which is suitable for purposes of preparing calibration standards for this type of instrumentation) which is mutually soluble with water such as, for example, a generally lower molecular weight alcohol or ketone, such that it may be easily evaporated at room temperature. Preferably, these lower molecular weight alcohols have three carbons or less, including methanol, ethanol, propanol, isopropanol, while acetone is the preferred ketone. In a particularly preferred embodiment, isopropanol is used.

Generally speaking, the stock solution and solvent may be mixed to prepare the treatment solution in any ratio which enables the salts to remain in solution and which is sufficient to enable the proper metallic contaminant concentration to be substantially uniformly deposited upon the reference wafer surface. Typically, however, the ratio of stock solution to solvent in the treatment solution ranges from about 1:1 to about 1:100, in terms of a volume ratio.

Once the stock solution and the solvent have been thorough mixed, and thus the treatment solution prepared, a wafer which is to be substantially uniformly contaminated in order to prepare the reference wafer is contacted with the treatment solution. Typically, the wafer is treated by immersing the wafer, either entirely or partially with wafer rotation being employed to ensure thorough treatment of the entire surface, for a time sufficient to ensure complete surface treatment and also to ensure the proper level of contamination is imparted substantially uniformly onto the entire wafer surface. Typically, therefore, this means that the wafer surface is contact with the treatment solution for about 1 minute to about 10 minutes.

Generally, the wafer to be contacted with the treatment solution may be any wafer suitable for purposes of preparing a calibration stand of this type, which have been prepared under stringent manufacturing conditions. Stated another way, essentially any silicon wafer may be treated provided it has first been subjected to cleaning processes standard in the art which is sufficient to effectively lower the concentration of metallic impurities of interest to a level which is sufficiently low, such that these impurities will not interfere with the calibration process. Accordingly, a "virgin" wafer, or a wafer having a substantially metal-free surface, will be used.

Once the treatment time has expired, the wafer is removed from the solution (i.e., excess treatment solution remaining on the wafer surface is removed) and then the wafer is dried in a conventional clean room setting (i.e., a room which is essentially free from any metallic contaminants which may interfere with the calibration process) at room temperature (i.e., at

temperatures typically ranging from about 20 to about 25°C). Due to the volatility of the treatment solution imparted by the organic solvent, the treated wafer will quickly dry without the need of employing means for speeding the drying process.

The dried wafer may now be utilized as the reference wafer for purposes of calibrating a given instrument, which is to be employed to analyze the surface of sample wafers obtained from the manufacturing process, following commonly practiced calibration methods for that particular instrument. In a preferred embodiment, the reference wafers of the present process may be utilized to calibrate a total X-ray reflection fluorescence instrument (such as, for example, model TREX610T, commercially available from Technos Corp., Inc. of Osaka, Japan) by means common in the art.

In view of the foregoing, it can be seen that in accordance with the process of the present invention, a reference wafer having a concentration of one or more metallic contaminants of interest is prepared, the concentration being distributed substantially uniformly over the entire wafer surface. The present process thus provides a more efficient and cost-effective means by which to prepare such wafers because it may be performed on-site, where wafer manufacturing occurs. Furthermore, by performing the present process at the wafer manufacturing facility and utilizing a wafer from the production line for purposes of preparing the reference wafer, more accurate and reliable data can be collected.

As illustrated by the following Example, a reference wafer suitable for the calibration of a total X-ray reflectance fluorescence instrument can easily and efficiently be prepared to have a contaminant concentration which is substantially uniform over the

entire wafer surface. However, it is to be noted that the process conditions provided herein, as well as those provide above, may be other than described without departing from the scope of the present invention.

EXAMPLE

Aqueous stock solutions of iron, copper, nickel, zinc, chromium, and calcium were prepared such that the concentration of each metal in solution was about 10 ppm. Aliquots from each stock solution were taken, ranging from about 1 to about 200 cc each, to which was added isopropanol such that the total volume of each solution was about 1000 cc. Each treatment solution, thus prepared, was shaken to ensure thorough mixing and then placed in an immersion tank. A well-cleaned six inch silicon wafer (cleaned by means standard in the art) was then immersed in the tank of the nickel-containing treatment solution for about 3 to about 4 minutes. The wafer was then removed from the tank and, after allowing the excess solution to drip therefrom, the wafer was dried at room temperature in a clean room.

After being dried, the reference wafer was then analyzed in nine difference locations on the wafer surface by means of a total X-ray reflection fluorescence instrument (TREX610YT model, from Technos Corp.). Referring now to Fig. 1, a diagram in the shape of a histogram is shown, illustrating the nickel deposits, integrating the results from all 9 locations. In addition, referring now to Fig. 2, the nickel deposits were also determined by repeating the noted procedure on a commercially available reference wafer which was prepared by spin coating.

The metal deposits were determined by the total reflection fluorescent X-ray apparatus made by Technos

Corporation, Inc. on the reference specimen prepared by the method as described above and on the commercially available reference specimen prepared by spin coating. The test resulted are shown in Table 1 below.

TABLE 1

Analyzing Point No.	Method of Present Invention	Commercially Available Specimen
1	13.0441	77.4742
2	8.0963	89.8240
3	11.5328	81.2665
4	11.8583	85.5087
5	11.2154	118.6100
6	10.5565	75.6795
7	7.4390	71.8325
8	9.6834	99.7520
9	8.6127	83.1578
Average Value	10.2265	87.0118
Standard Deviation	1.8983	14.4421

As evidenced by these test results, it can be observed that the nickel deposits of the reference wafer prepared by the present invention are substantially uniform, the uniformity of the nickel contamination being significantly improved relative to the commercially available reference wafer. Thus, it has been found that the reference wafer of the present invention is acceptable for purposes of industrial use, the present process therefore being suitable for preparing calibration wafers for use in quantitative analysis.

It is to be noted that the metal deposits were practically determined using the reference specimens of the present invention, and the obtained determination results were found to be high in reproducibility.

As described above, zinc, nickel, copper, iron, calcium and chromium, for example, may be deposited in a predetermined amount to a virgin wafer surface in order to prepare a reference specimen, by means of preparing an aqueous salt solution and mixing it with a specific organic solvent. The so prepared reference specimen is thus used in the determination of metal deposits on the surface of a sample wafer by means of a total reflection fluorescent X-ray apparatus. In this way, the present invention can exhibit the effect that the metal deposits on the wafer surface can be determined more accurately and the product inspection of the finish wafer, quality control and the like at the production sites can be performed with ease.

In view of the above, it will be seen that the several objects of the invention are achieved.

As various changes could be made in the above processes without departing from the scope of the invention, it is intended that all matter contained in the above description be interpreted as illustrative and not in a limiting sense.



What is claimed is:

1. A process for preparing a reference wafer suitable in the calibration of a total reflective X-ray fluorescence spectrometer, a surface of the reference wafer having a substantially uniform distribution of a metallic contaminant deposited thereon, the process comprising:

preparing an aqueous salt solution comprising water and a water-soluble salt of a metallic contaminant;

mixing a portion of the aqueous salt solution with an organic solvent to form a treatment solution;

immersing a substantially metal-free silicon wafer in the treatment solution;

removing the treated wafer from the treatment solution; and,

drying the treated wafer.

2. A method for mapping a metal deposit distribution on a sample wafer by means of a total X-ray reflection fluorescence instrumentation, the method being characterized in that a reference wafer which has been prepared by immersing a virgin wafer for a predetermined period of time in a mixture obtained by mixing water-soluble salts of desired metallic contaminants with an organic solvent selected from a lower alcohol or a ketone and dried is utilized in the calibration of said instrument.

3. A method according to claim 2, wherein isopropanol is used as the organic solvent.

This Sheet Has Been Replaced

