Journal of Electron Spectroscopy and Related Phenomena 178–179 (2010) 433–448

Contents lists available at ScienceDirect

Journal of Electron Spectroscopy and Related Phenomena

journal homepage: www.elsevier.com/locate/elspec

XPS and angle resolved XPS, in the semiconductor industry: Characterization and metrology control of ultra-thin films

C.R. Brundle[a,*], Giuseppina Conti[a,1], Paul Mack[b]

[a] C.R. Brundle and Associates, 4215 Fairway Drive, Soquel, CA 95073, USA
[b] Thermo Fisher Scientific, The Birches Industrial Estate, Imberhorne Lane, East Grinstead, West Sussex, RH19 1UB, United Kingdom

ARTICLE INFO

Article history:
Available online 12 March 2010

Keywords:
XPS
ARXPS
Semiconductor wafer
Metrology
Materials characterization

ABSTRACT

This review discusses the development of X-ray photoelectron spectroscopy, XPS, used as a characterization and metrology method for ultra-thin films in the semiconductor wafer processing industry. After a brief explanation of how the relative roles of XPS and Auger electron spectroscopy, AES, have changed over the last 15 years or so in the semiconductor industry, we go into some detail as to what is implied by metrology, as opposed to characterization, for thin films in the industry, and then describe how XPS, and particularly angle resolved XPS, ARXPS, have been implemented as a metrology "tool" for thickness, chemical composition, and non-destructive depth profiling, of transistor gate oxide material, a key requirement in front-end processing. We take a historical approach, dealing first with the early use for SiO_2 films on Si(1 0 0), then moving to silicon oxynitride, SiO_xN_y in detail, and finally and briefly HfO_2-based material, which is used today in the most advanced devices (32 nm node).

© 2010 Elsevier B.V. All rights reserved.

Contents

1. Introduction 433
2. Characterization and metrology 435
   2.1. Characterization 435
   2.2. Metrology 435
   2.3. Precision and process tolerance 435
3. Development of XPS for characterization and metrology of ultra-thin films 436
   3.1. Background 436
   3.2. SiO_2 gate oxide film thickness as the prototype example 436
   3.3. SiO_xN_y gate oxide films 438
      3.3.1. Background 438
      3.3.2. Initial attempts at thickness and N dose determination for SiO_xN_y 438
4. Depth profiling by XPS in general and the determination of the N profile in SiO_xN_y in particular 440
   4.1. The RDP method 440
   4.2. Modeling the ARXPS response 441
   4.3. Simultaneous thickness, N dose, and N distribution wafer metrology by ARXPS 447
5. Beyond SiO_xN_y 447
6. Conclusions 448
   Acknowledgements 448
   References 448

1. Introduction

This review covers the development of XPS within the semiconductor wafer processing industry for materials characterization and materials processing metrology. Specifically, it concentrates on the use of Angle Resolved XPS, ARXPS, for the characterization and metrology of ultra-thin films. As such, much of what is discussed

* Corresponding author. Tel.: +1 8314791399.
E-mail address: brundle@attglobal.net (C.R. Brundle).
1 Formerly at Applied Materials, Inc., Santa Clara, CA, USA.

0368-2048/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.elspec.2010.03.008

here could also be applicable to the disk drive data storage industry, where ultra-thin films appeared in manufacturing processes before they did in semiconductor technology, and in any other industry where control of ultra-thin films is needed. What these "high tech" industries have in common then, is that they all involve growth or etching of films, which have to be well-characterized and precisely controlled. The uses of XPS here are in the areas of process development, process control (particularly metrology), quality assurance, and failure analysis.

What this review is not about, but about which it is necessary to say a few words, is the traditional use of XPS as a surface analysis tool in the chemical, chemical engineering, and metallurgical industries. XPS has been used in these industries, such as oil refining, fine and heavy chemicals, and polymers, ever since it was originally established as being highly surface sensitive some 40 years ago [1]. For many years, however, its role was largely confined to research and development areas, where XPS capability might be found as a part of the traditional "materials laboratory". The XPS instrumentation in the materials laboratory at that time did not differ significantly from that in academia, and its effective use required a similar level of specialized expertise. Owing to its extreme surface sensitivity, its chemistry speciation capability, and its relatively easy quantification for surface species (*note*: not necessarily for bulk, or for a non-uniform depth distribution), which were all recognized early on [2], XPS naturally became used where the needs for these attributes was undisputed, such as surface contamination issues (leading to adhesion or corrosion failures, for instance), surface reactions and poisoning in heterogeneous catalysis, and the modification or degradation of polymer surfaces. XPS instrumentation however, had essentially no lateral resolution, and depth profiling was both awkward and slow to perform (alternate cycles of Argon sputtering and spectral acquisition), and often introduced artifacts of change in chemistry [3]. These two factors, no lateral resolution and slow depth profiling capability are part of the reason that XPS did not gain much early traction in the semiconductor device production industry, whereas e-beam Auger electron spectroscopy, AES, did. E-beam AES had sufficient lateral resolution to work at the device level, primarily for failure analysis [4]. This and the seemingly mundane attribute that elemental depth profiling (no chemical state information, and therefore no obvious evidence of the possible sputter induced chemical changes) was done rapidly through micrometer thick films, were very influential in the early adoption of AES in the semiconductor industry materials labs. Also the materials used then in the industry were rather simple and relatively inert (Si, SiO_2, Al, W), meaning that chemical speciation was of lesser importance.

So, from 40 years ago to about 15 years ago, XPS was more widely used for true surface issues in the chemistry-based industries, primarily in the R&D laboratories, whereas AES was not (charging of insulators, no chemical state information as practiced, poor quantification). AES was used more widely in the semiconductor industry and other industries where thin films were deposited as a basis of the technology. AES was not being used primarily for real surface analysis though and certainly not for chemistry. It was primarily a qualitative thin film elemental profiling tool, with excellent lateral resolution, if needed.

Over the last 15 years or so this balance has changed gradually, so that now XPS is much more used in the thin film arena, though, of course, for high spatial resolution, AES must still be used. The main driving force for this change has been the change in the semiconductor industry itself. There has been a steady decrease in the thickness of many layers termed "thin films" down to, in some cases, sub-nanometer, which has made XPS a highly suitable technique for materials characterization of the whole film, because of the match in thickness to XPS probing depth. In addition the surface of a layer, or interface between layers, on the nanometer scale was previously often technologically irrelevant when film thickness approached micrometer dimensions. A few Angstrom of reaction was of no concern for the bulk properties of the film (though it could be a practical showstopper because of delamination for instance). Today, with many films of only nanometer's thickness, the surface/interface reaction may consume a considerable fraction of the film and strongly affect the overall film properties. This is exacerbated by the recent trend to more exotic and reactive materials. The instability and reactivity of HfO_2 at a Si interface, where processing conditions can result in interdiffusion and a complex phase mixture, is a perfect example – yet HfO_2-based sub-20 Å films are in current production as the gate dielectric material in the most advanced transistors, and therefore must be very precisely controlled. Another example is the switch from Al to Cu for the interconnect metallurgy. Cu diffusion into the active device region is catastrophic to device performance, which leads to the need to introduce an additional effective barrier layer, such as Co. This, however, can only be a few nanometers thick, or the conductivity benefit of moving from Al to Cu for reduced dimension structures is compromised (the barrier layer takes up thickness that should be available to the Cu interconnect wire). Likewise, there are issues of controlling a few Angstrom of oxidation on the Cu seed layer which is deposited prior to electroplating the interconnect lines. The dielectric insulation between the interconnect levels has also changed drastically, and there may be up to nine interconnect levels in advanced product. Traditionally the insulation was SiO_2. Now exotic organic/inorganic material mixtures are also used, which can have homogeneity and stability issues (towards humidity, for instance).

Summarizing, the industry has evolved to the point where:

(a) Layers can be so thin that there is little distinction between a surface or interface region and the bulk of the film,
(b) XPS is an appropriate approach to analysis for the whole film thickness, and
(c) The wide range of materials now used requires much more chemistry understanding and control.

Hence the growth of XPS as an analytical tool, not (just) for surface issues, but for the whole film.

While the above changes were occurring in the semiconductor industry, the XPS instrumentation was also evolving. Lateral resolution has gradually improved for lab-based equipment to the point where it is quite feasible to work on the scribe lines and test areas between dies on patterned wafers, thus allowing measurement at any stage during the wafer processing steps. This requires current lateral resolution of about 10 μm, with enough signal strength to allow measurement in minutes rather than hours. Of course current lab XPS equipment does not (and probably never will) have the capability to work at the device structure level, where 10s nm resolution is needed.

The instrumentation intended for use in the industry also started seriously diverging in design from that intended for general purpose usage about 15 years ago. Instruments capable of taking large samples, and eventually full 200 mm and 300 mm wafers became available. They also became more automated, initially in terms of loading wafers and acquiring data in different locations (mapping), and later in automating the data reduction process to produce what might be termed the XPS analyst's "standard table"; that is a table of percentage elemental compositions, based on Scofield's atomic photoionization cross-sections [5], or empirically determined cross-sections for the particular instrument design, plus some information on chemical speciation of the elements present from resolved chemical shifts. Thus, the need for expert involvement to get to this stage – the standard table – was greatly reduced and therefore access to the technique greatly widened.

Download English Version:

https://daneshyari.com/en/article/5396662

Download Persian Version:

https://daneshyari.com/article/5396662

Daneshyari.com