Growth mechanism of iron nanoparticles on (0001) sapphire wafers

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Abstract

Laser ablation of a high purity (99.7\%) iron target was used to accomplish the depositions of iron nanoparticles on the (0001) face of single crystal sapphire wafers. The nanoparticles were characterized in situ by means of X-ray photoelectron spectroscopy (XPS). The growth mechanism was determined by applying the QUASES-Tougaard methodology to the extended part of the background intensity of the Fe KMM peak in XPS spectra. The heights of nanoparticles obtained are between 3.5 and 6.5 nm. In the first 150 laser pulses, the height of the nanoparticles remained constant while the coverage was increased.

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1. Introduction

The study of nanoparticles is a subject of high importance in the area of material science, due to the potential applications in catalysis, magnetic recording media, and more recently in medicine. When a material is in the form of nanoparticles, it shows properties different from those of the same material in the form of thin film or bulk. Those properties are strongly influenced by the size of the nanoparticles. The size of the nanoparticles depends on the preparation technique and thereupon on the deposition parameters. Physical methods are scarcely used to grow nanoparticles but they are very effective in the growth of nanoparticles over a substrate. Among the physical methods used is laser ablation, which allows the deposition of nanoparticles with control down to the atomic scale. On the other hand, it is necessary to know if deposited materials are free of contaminations, so it is necessary to determine the chemical composition of the nanoparticles with a technique such as XPS. The aim of this work is to show that XPS can be used to obtain quantitative information of the nanoparticles, such as coverage, height and the chemical quality of the deposited nanoparticles.

2. Experimental

Fe nanoparticles were deposited in a commercially available Riber LDM-32 Laser Ablation System, equipped with in situ XPS. The deposits were carried out with a Kr–F excimer laser (\( \lambda = 248 \) nm, with 20 ns pulse width). All deposits were made using the same laser condition, namely 200 mJ per pulse, a frequency of pulses of 2 Hz and a laser fluence of 0.7 J/cm\(^2\) at the target surface.

Each deposit was made following different steps; each step consisted in the deposition of a certain amount of iron and immediately after, the deposition was characterized in situ by XPS, at the end of each step; that deposit is used as substrate for the next step. The total number of laser pulses used in this deposition was 400. A number of samples were repeated on dimpled substrates, in order to observe the nanoparticles by transmission electron microscopy. Those results are not shown here.

For the XPS background analysis, windows of 300–800 eV in kinetic energy (Fe Auger peak and Fe 2p peak) of the spectra were used. The background for each...
spectrum was analyzed using the QUASES [1] methodology and software.

3. Results and discussions

Fig. 1 shows the flux density of photoelectrons excited from a single Fe atom (full line), denoted by \( F(E_0, \Omega) \); this density is obtained after subtracting the background (dotted line) to the XPS spectrum of a thick Fe film (dashed line). All XPS spectra taken of deposited nanoparticles were processed with the QUASES-Tougaard methodology. According to this, the Fe concentration at depth \( x \) is varied until each spectrum match with \( (F, \Omega) \). When that match is reached, the atomic concentration is correct, that is, the coverage and the size of the nanoparticles have been determined.

Fig. 2 shows the height of nanoparticles as a function of the number of pulses (left side) and of the coverage (right side). In this figure, we can observe that for the first 150 pulses the height of nanoparticles changed from 3.5 to 4 nm while the coverage was incremented up to 80%. That result indicates that during the first 150 pulses, the material deposited was spread over the substrate surface, conserving the height of nanoparticles. After 150 pulses, the new material grew over the nanoparticles previously deposited, increasing the height and coverage. When the deposit was made using 400 laser pulses, the coverage is near to 100% and the height of nanoparticles increased up to 6.5 nm. The amount of material deposited (defined as the product of height of nanoparticles with its respective coverage for a specific deposit) is proportional to the number of the pulses, that is, more laser pulses during the deposit imply more material on the substrate.

4. Conclusions

Iron nanoparticles were deposited by pulsed laser ablation; these nanoparticles grew with a constant height of approximately 4 nm during the first 150 laser pulses. In this pulse range, an increment in the coverage was observed, indicating that we had more material on the substrate. After 150 laser pulses, both coverage and height of nanoparticles increased their values to 6.5 nm and 99%, respectively.

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References


Fig. 1. Flux density of photoelectrons excited from a single Fe atom (full line), background (dotted line) and XPS spectrum of a thick Fe film (dashed line).

Fig. 2. Height of nanoparticles as a function of number of pulses (left side) and coverage (right side).