話題

Sample Rotation in Sputter Depth Profiling

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1. INTRODUCTION

An important step forward in improving the depth resolution in sputter depth profiling was the introduction of sample rotation by A. Zalar in 1985 /1/. Since then, numerous papers using depth profiling with sample rotation in combination with AES, SIMS and XPS have appeared which showed improvement of the precision and accuracy of sputter depth profiles. In order to provide a reliable judgement on the capabilities and limitations of the sample rotation approach top depth profiling, an interlaboratory comparison between four laboratories using these three techniques and specified sputtering conditions was organized in 1993 /2/. Today, sample rotation is a well established technique and is supplied by any of the major manufacturers of surface analysis instruments. Despite this fact, a general physical theory of the effect of sample rotation is still missing. This paper tries to understand and generalize the main observations of different researchers. For further details the reader is referred to /3,4/.

2. BASIC PRINCIPLES OF OPERATION

The basic idea of sample rotation during sputtering came from the idea that unidirectional ion incidence, particularly at certain angles around 35 degrees in textured thin films, led to strong degradation of the depth resolution because of the different orientations of the small grains. In order to wipe out these directional differences, a multidirectional ion incidence should be used. In practice, however, this is very difficult to realize; therefore by rotating the sample under an inclined ion beam one at least has a 360 degree variation of the azimut angle whereas the meridian (or polar) angle remains fixed. Thus sampel rotation only is a sequential exposure of the sample to all azimutal angles and therefore is a crude approximation of the desirable instantaneous multidirectional exposure.

It should be kept in mind that the influence of sample rotation on depth resolution is limited to those features which are caused by variations of local sputtering rates within the analyzed area and it does not include the fundamental ion-beam/sample interaction effects such as atomic mixing and surface roughening on the monolayer scale due to sputtering statistics /5/. Local sputtering rate variations depend e.g. on differences in lattice orientations in polyceystalline samples and on local differeces in the inclination angle of the microplanes to the ion beam in case of rough surfaces, and on inhomogeneous ion beam intensities. The importance of these parameters in optimization of depth profiling with sample rotation is briefly discussed in the following.

3. OPTIMIZATION OF IMPORTANT PARAMETERS

There are some cases, where sample rotation brings little or no improvement, e.g. in depth profiling of amorphous samples with noble gas ions. This includes semiconductors or oxide layers because they were already produced in an amorphous state or they are transformed into an amorphous state by ion bombardment. Due to the nondirectional metallic bond, the crystalline state in metals and alloys is much more stable, even during sputtering.

3.1 Influence of Lateral Ion Current Density Variations

The effect of laterally nonuniform ion beam intensity distribution on the depth resolution is considerably reduced by sample rotation /6/. However, it was shown that only the linear component of the lateral dependence is compensated but not the quadratic and higher order components /4/. Therefore it is essential to work with a rastered ion beam and in the center (away from the edges) of the sputter crater, and the analysis area should be small against the raster dimensions /7/. Any radial inhomogeneity in the analyzed area, e.g. by neutrals in the ion beam, by electron induced

desorption /8/ or an excentric rotation axis combined with nonlinear ion current terms /4,7/ is observed as an oscillatory behaviour in the measured profile and as a linear degradation of the depth resolution with sputtering time /4/.

3.2 Adjustment of the Analyzed Area

If the rotation axis is not centered in the analyzed area, the latter changes with the angle of rotation. While for a completely homogeneous sample and ion beam this should not make any difference, in practice it leads to periodic distortions which again pile up with time /4/. Adjustment can be done by zooming in with an optical or SEM imaging device. In SAM, any special feature can be used. A ring-shaped pattern formed by electron beam generated residues of the contamination layer is often used to exactly find the rotation axis. A certain point away from the rotation axis can only be analyzed if there is an automatic readjustment of the position to that of the analyzed spot after each small increment of rotation /9/.

3.3 Angle of Incidence of the Ion Beam

Experimental /2,4,10/ and theoretical /3/ studies agree in the following observations: At zero degree icidence (i.e. normal to the sample surface) of course there is no effect, at about 30 degree the effect is remarkable and the depth resolution steadily improves with increasing incidence angle, and above 60 degree improvement is almost vanishing depending on the original surface roughness.

The depth dependence of depth resolution in AES sputter profiling of Ni/Cr multilayer samples consisting of a total of 16 alternating Cr and Ni layers with a single layer thickness of 30 nm with 3 keV Argon ions at 45 and 70 degree incidence angle was studied in /2/ with and without rotation and for two different rotation speeds. For these metallic samples it is typical that without rotation the depth resolution delta z increases with sputtered depth due to the crystalline orientation effect /3, 11/. In contrast, independence on depth is obtained by using sample rotation. For smooth samples, delta z decreases with higher incidence angle with and without rotation . The optimum value of delta z of about 3 nm at 70 deg. incidence angle and high rotation speed corresponds approximately to the physical limit of the atomic mixing zone width for 3keV Ar+ ions /12/. In contrast to samples with smooth surfaces, stationary profiling of samples with rough surfaces causes an increase of delta z for ion incidence angles above 60 degree, depending on the angular distribution of the microplanes /13/. With sample rotation, in particular the shadowing effect encountered in unidirectional sputtering is considerably reduced. However, it is obvious that at very high incidence angles the deeper troughs cannot be reached even by sample rotation and therefore a - less pronounced - increase in delta z is predicted and observed /2,

3.4 Speed of Rotation

Under optimized conditions, the extent of ion beam induced roughening in metallic layers is mainly determined by the dependence of the sputtering rate on the orientation of the different grains in the sample /3,11/. The incremental amount of growth of a surface roughness R depends on the difference of the maximum and minimum sputtering rate for non-channeling and channeling directions and on the dwell time of the ion beam in the vicinity of a certain angular distribution around a channeling direction /2,3/. This dwell time is proportional to the rotation speed.. The decisive parameter is not its absolute value, but the sputter rate/ rotation speed ratio , and R is proportional to this ratio.

A lower limit for a useful rotation speed is estimated as follows: For one completed revolution the roughness development R cannot be larger than half the average sputtered depth. for example, allowing a maximum of 1 nm for R this means that an optimum rotation speed of about 1 rpm (revolutions per minute) is sufficient for a sputtering rate of about 2 nm/min. Values of 2....10 for the above ratio of sputter rate/rotation speed have indeed been found adequate in many experiments /2,4/. Of course the above criteria depend on the amount of roughness contribution relative to other contributions, e.g. atomic mixing or original surface and/or interface roughness to the total,measured depth resolution /5/. In a systematic study, Tanemura et al./14/ have demonstrated the influence of the rotation speed on depth resolution, and their results could be semiquantitatively explained in ref. /2/.

REFERENCES

/1/ A. Zalar, Thin Solid Films 124 (1985) 223

/2/ S. Hofmann, A. Zalar, E.-H. Cirlin, J.J. Vajo, H. J. Mathieu and P. Panjan, Surf. Interface Anal. 20 (1993) 621
/4/ S. Hofmann and A. Zalar, Surf. Interface Anal. 21 (1994) 304
/3/ W. Hoesler and W. Pamler, Surf. Interface anal. 20 (1993) 609
/5/ S. Hofmann, Progr. Surf. Sci. 36 (1991) 35
/6/ E.-H. Cirlin, Thin Solid Films 220 (1992)197
/7/ R. E. Ericson, Surf. Interface Anal. 18 (1992) 381
/8/ A. Zalar and S. Hofmann, Appl. Surf. Sci. 68 (1993) 361
/9/ J. Geller, priv. communication.
/10/ M. M. Henneberg, D. J. Pocker and M. A. Parker, Surf. Interface Anal. 19 (1992) 55
/11/ G. Ecke, T. Woehner, H. Roessler and S. Hofmann, Proc. ECASIA 95, Wiley, Chichester 1996, p.587
/12/ S. Hofmann, Appl. Surf. Sci. 70/71 (1993) 9
/13/ A. Zalar and S. Hofmann, Nucl. Instr. Meth. Phys. Res. B18 (1987) 655
/14/ M. Tanemura, S. Aoyama and F. Okuyama, Surf. Interface Anal. 18 (1992) 475