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AUGER MICROPROBE TEMPERATURE PROFILES OF CONTAMINATION RESIDUE
ON SINGLE CRYSTAL FERRITE SUBSTRATES

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Abstract

Single crystal manganese-zinc ($\text{MnO}, \text{ZnO}, \text{Fe}_2\text{O}_3$) ferrite wafers with a 50 Angstrom² surface finish were subjected to a conventional cleaning procedure and then analyzed in a JAMP-10 Auger microprobe with an internal heating device. Three essential Auger peaks were monitored during the heating. Auger peak intensity vs. time profiles were obtained for the heating process. High vacuum heat treatment conditions were established for the preparation of these materials for use as substrates in UHV thin film growth.

Introduction

Substrate preparation is believed to be critical for the formation of epitaxial relationship between a deposited film and the underlying substrate material. The surface of the substrate must be polished to a mirror-like finish and be free of contaminants such as organics and metal ions. Most cleaning procedures for substrates used in epitaxial growth involve a degrease and a chemical etch which may or may not be followed by a heat treatment in a vacuum chamber. An abundance of literature exists on the cleaning procedures for semiconductor [1, 2] and garnet [3] substrates, but not for those consisting of manganese-zinc ferrite. In this study, we chose to approach the problem of ferrite cleaning by employing a standard degreasing sequence and a UHV bake.

Experiment Description

The following Auger microprobe study was designed to verify the cleaning procedure used for substrates subject to thin film growth. The heating stage attachment which was installed on the JEOL JAMP-10 Auger microprobe was, according to the vendor, to provide a temperature range of from room temperature up to 600°C. During the course of this experiment, however, we were not able to reach temperatures in excess of 550°C. The temperature of the heating element was controlled by manually adjusting the current to the heating stage while monitoring the thermocouple electromotive force EMF.

The single crystal ferrites were purchased from three vendors (Fuji, JVC and Shinetsu) and they were then

Key Words: Auger electron spectroscopy, substrate cleaning/preparation, manganese-zinc ferrite, heating stage attachment

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polished by another vendor to a 50 Angstrom surface finish. A wafer from each of the suppliers was prepared for the Auger experiment by ultrasonically cleaning it in acetone for 5 min. and in methanol for 5 min., and by rinsing it in DI water. Additionally, one of the substrates was subjected to a longer acetone treatment (20 min).

Auger analysis was performed on all four samples in order to determine what heat treatment would adequately remove solvent residues from the degreased ferrite surface. The Auger analysis was performed at 0.2 microAmps absorbed beam current. Defocussed electron beam of about 30 microns diameter was used to minimize the effect of the beam on decomposition and desorption of organic species. Initial Auger survey spectra exhibited significant carbon peaks. The intensity of the carbon Auger peak at 270 eV was used as an indicator of the presence of the residue, and oxygen and iron Auger peaks at 508 eV and 703 eV, respectively, represented the substrate material during the heat treatment experiment. (Oxygen peak may have also been an indicator of the hydrocarbon residues, but it could not be used as such because of the oxide nature of the substrate).

Standard depth profiling software was used to perform the peak intensity measurements through the computer at preset time intervals. By changing the sample temperature at specific points in the process, we were able to obtain peak intensity vs. time profiles as a function of temperature for all four samples (Figs. 1-4).

In the actual heating process, the sample temperature was first increased from room temperature to 315°C and held at that level for about 12 minutes, after which it was again increased to 495°C where it was maintained for 15 minutes. The final temperature of the heat treatment was 550°C. The sample was kept at this temperature until the carbon peak intensity showed a significant decrease. After the heating profiles were completed, final Auger survey spectra were collected on each sample to assure that the heating resulted in the removal of the organic residue from the surface of the samples (i.e. the Auger carbon peak

intensity dropped below 2% of the initial value).

Results/Discussion

The Auger analysis indicated that no significant changes were observed in the carbon peak intensities on any of the samples during the 12 minutes of heat treatment at 315°C. At 495°C, the carbon peak intensity on the samples cleaned for 5 minutes in acetone decreased at a steady rate of about 1.5% of relative intensity per minute. The decrease in the carbon signal was even greater at 550°C -- for the Fuji and Shinetsu wafers the rate was 2-3% per minute, while that for the JVC wafer was 4.5% per minute (See Fig. 2).

A significant change in the behavior of the carbon peak intensity was observed on the second Shinetsu sample which was subjected to a 20 minutes acetone treatment during the cleaning procedure. For this sample, the carbon peak indicated decrease shortly after the temperature was brought to 495°C. The rate of decrease was sharp enough (2% per min.) that after 15 minutes of treatment at 495°C, the intensity of the carbon peak was down to almost zero - about 2% relative intensity (compare plots #3 and #4 of the two Shinetsu substrates to see the effect of modifying the degreasing procedure). It seems that the longer acetone rinse removes more hydrocarbon residue from the wafer, so the remaining residue can be desorbed at a temperature where the carbon removal rate is lower. This result can be very useful in establishing the cleaning procedure for the substrates -- it indicates that a higher temperature heat treatment (550°C) prior to the UHV thin film deposition may be substituted by a longer (up to 20 minutes) acetone rinse followed by a lower temperature vacuum bake.

It should be noted that the temperatures indicated in this study were measured by a thermocouple which was positioned under the sample, so that the temperature measured by the millivoltmeter might have been slightly different from that of the sample itself. This situation is quite typical in thermocouple temperature measurements and it should be taken into consideration when the conditions for the substrate UHV in-situ thermal cleaning are established.

Auger Microprobe Temperature Profiles of Contamination

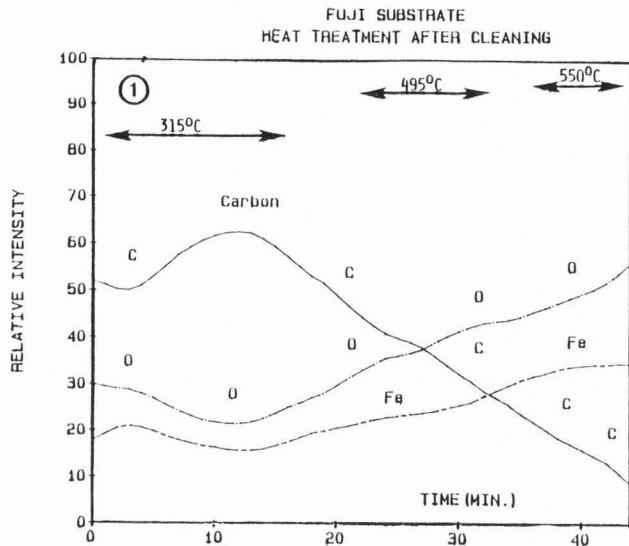


Fig. 1. Auger temperature profile for Fuji substrate after standard cleaning procedure (5 min. in acetone + 5 min. in methanol).

Fig. 2. Auger temperature profile for JVC substrate after standard cleaning procedure.

Fig. 3. Auger temperature profile for Shinetsu substrate after standard cleaning procedure.

Conclusion

A proposed cleaning procedure for single crystal Mn-Zn ferrite substrates was examined by means of an Auger microprobe fitted with a heating stage. Using standard depth profiling software, we were able to obtain peak intensity vs. time profiles as a function of temperature for both the organic residue and the substrate elements. We conclude that a standard solvent degrease followed by a vacuum bake at 495-550°C is sufficient to remove impurities from the ferrite wafer so that it may be used as a substrate for epitaxial growth.

References

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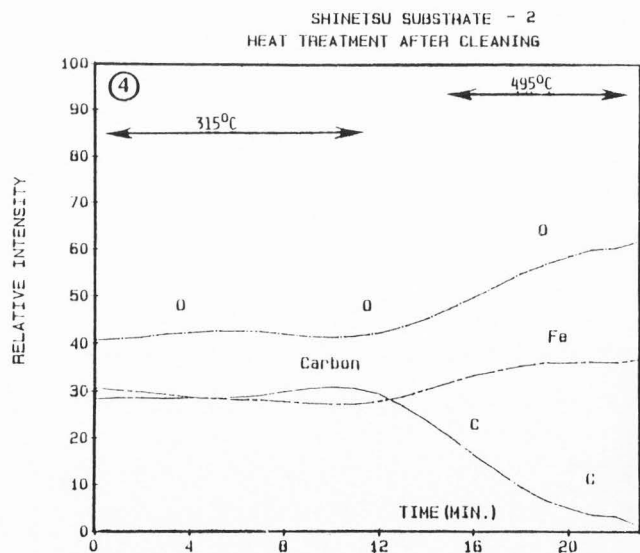
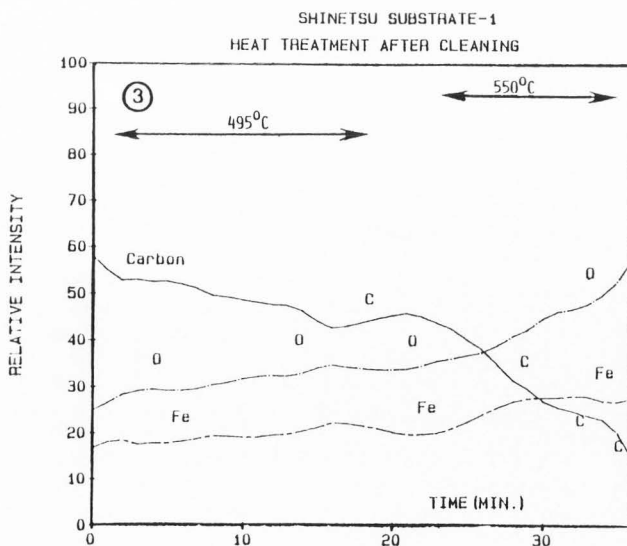
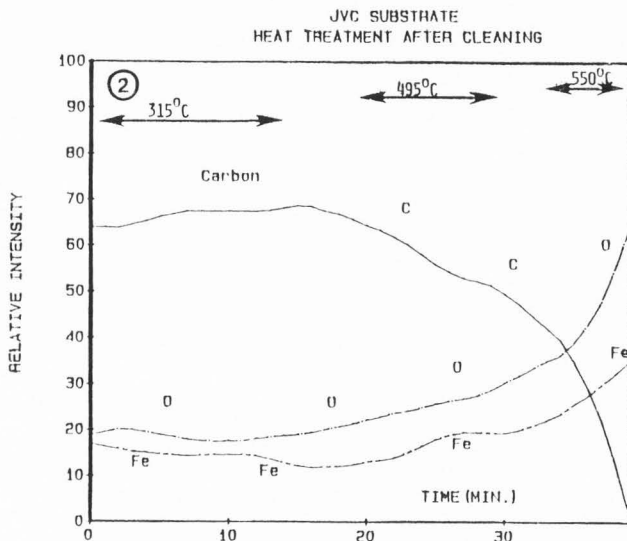


Fig. 4. Auger temperature profile for Shinetsu substrate after modified cleaning procedure (acetone treatment for 20 min.) Compare with Fig. 3 for the same substrate.

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Discussion with Reviewers

G.G.Hembree: The duration of the heating periods should be more clearly indicated on the graphs.

Authors: In each experiment there was a temperature ramp between the two temperatures indicated on the graphs. The Auger peak measurements were continuous, whereas the temperatures are indicated at the times when the temperature reached the level shown on the graphs.

G.G.Hembree: The basis of the maximum carbon relative intensity on the graphs is not clear. Is it the value of the carbon Auger peak before heating or just an arbitrary level?

Authors: The basis for the carbon peak intensity was the carbon relative intensity measured at the time when the temperature reached the value indicated on each graph as a starting temperature.

G.G.Hembree: What is the significance of the 2% of initial value level? Does this criterion indicate that less than a fraction of monolayer of carbon exists on the surface? Your claim that the procedure you describe is sufficient to remove all impurities seems to be highly optimistic in light of the known insensitivity of Auger spectroscopy to low concentrations of elements.

Authors: We arbitrarily used the 2% value for the carbon peak intensity based on the consideration that carbon content below that level could not be detected by this method. We do not claim that all impurities were removed by the combination of cleaning and heating procedures. We indicated only that the heat treatment resulted in the increased rate of removal to the point when carbon peak intensity was reduced below detectable level.

G.G.Hembree: Why did you choose to use acetone as your primary solvent? Acetone is known to leave residues which are very difficult to remove. Better degreasing procedures for UHV materials are outlined in "Methods of Experimental Physics", v.14, Vacuum Physics and Technology, Academic Press, New York, 1979, pp. 499-500.

Authors: Acetone is a common powerful solvent which is used in our processing. Obviously, the residues could be removed if acetone treatment is followed by methanol rinse. It was also shown that longer acetone treatment left a lower amount of surface contamination.

J.Leys: Was the instrument vacuum environment precisely the same for all of the experiments?

Authors: We monitored the vacuum conditions through the experiment. Obviously, the vacuum deteriorates with the heating inside the sample chamber.

J.Leys: Were the substrates all of the same thickness and did they have the same contact area with the heating stage?

Authors: Yes, the thicknesses were the same, and the heating stage could accommodate only a limited size sample, which was placed directly above the heating element. The samples were the same size.

J.Leys: Were any of the experiments repeated to determine reproducibility of the results?

Authors: Yes, some of the experiments were run more than once. They demonstrated reasonable reproducibility.

General comment: The substrates were given their final polish by the same vendor. We believe that the surface conditions after the final polishing process could depend upon the initial surface conditions of the substrates from three different vendors. In the course of this experiment we were looking for a trend in the contamination removal, rather than absolute numbers which were not critical for our applications. The values which we observed were different for the substrates from different vendors. However, we realize, that variations in the contamination removal rates could be attributed to the inaccuracies in the control of the heating conditions as well as to the difference in the initial surface conditions.

E.D.Jungbluth: A 20% increase in the carbon signal occurs in the 315°C interval, but only in the Fuji substrate. Can this be explained?

Authors: We explain this increase in the relative carbon signal at the beginning of the heating process of the Fuji substrate by the release of some absorbed surface contaminants which were not detectable at the room temperature. The difference in the surface conditions between different substrates can explain the fact that this event was not registered for other substrates.