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Behavior of (1 1 1) grains during the thermal treatment of copper film studied in situ by electron back-scatter diffraction

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Abstract

An annealed Cu blanket film was investigated in situ at high temperature using electron back-scatter diffraction (EBSD). The primary aim of the experiment was to study the changes in the (1 1 1) texture in the Cu film where the microstructure was already stabilized by previous annealing treatment. Two separate investigations were carried out at the same location of the film for better statistical reliability of data. It was found that the (1 1 1) planes got increasingly inclined to the specimen surface with increasing temperature. Additionally, a change in the strength of $\{111\}\langle110\rangle$ and $\{111\}\langle112\rangle$ texture components was observed with increasing temperature. Absence of these phenomena in freestanding Cu film indicates the impact of substrate on the behavior of (1 1 1) grains. The effect of substrate on the peculiar behavior of the (1 1 1) grains has been explained by a model which describes the contribution of both dislocations and diffusion to the observed phenomenon. The tilting of the (1 1 1) grains is discussed with reference to the recently reported Bauschinger effect in the Cu films.

Keywords: Cu film; Texture; OIM; EBSD; XRD; In situ

1. Introduction

After the replacement of Al by Cu as an interconnecting material, a lot of research has been done to understand the behavior of Cu interconnects and also Cu films [1–8]. The damascene process which was developed to make the incorporation of Cu in the electronic chip possible has undergone a lot of changes with respect to the new dielectric and barrier materials. A lot of data have been published showing the texture and microstructure evolution in the Cu lines and films as a function of new materials and deposition techniques. But still there has been almost no attempt made to understand the mechanisms responsible for texture and microstructure evolution especially in the Cu lines [9,10]. Such attempts to some extent have been made for the Cu films but still there are contradictions amongst the authors with regard to the role of stresses, grain growth mechanism and the role of impurity distribution [11–14]. The texture and microstructure control the reliability of the Cu lines and integrated circuit on the whole. It has been shown already that these parameters have a significant effect on stress voiding and electromigration [15–18]. But it becomes more important to comprehend the inherent tendency of the system to transform texture and microstructure if we are to shrink further the dimensions of the IC and make it more reliable. Such microstructural transformations are a strong function of internal and external parameters like trench aspect ratio, line width, passivation layer, annealing temperatures and time, residual stresses, impurities, etc. However, it should be mentioned that it is very difficult to explain explicitly the effect of these parameters on the texture and microstructure evolution in the Cu lines at this point of time when the behavior of Cu film itself is not very clear, though some recent attempts have been made for Cu films [19–22].

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Our earlier investigations were focused on the Cu interconnects wherein we studied the texture and microstructure variation as a function of line width, trench aspect ratio and annealing [23–26]. In order to understand the mechanism of texture and microstructure evolution in the Cu interconnects as a function of line width and annealing, we have attempted to study in situ high-temperature behavior of two different Cu films, one where microstructure was already stabilized by previous annealing and another where microstructure was still evolving at RT. This is because the texture and microstructure formation in the overburden Cu film during pre-CMP (chemical mechanical polishing) annealing considerably affects the texture and microstructure in the Cu lines beneath. Results for the Cu film where microstructure was still evolving at room temperature have already been reported [27]. The idea behind performing the in situ studies on the Cu films was to gain an insight into the changes occurring in the Cu interconnects during pre- and post-CMP annealing. Our previous results [27] help to understand the texture and microstructure evolution in the Cu interconnects during pre-CMP annealing. The investigation reported in the present article helps to understand the changes occurring in the Cu lines during post-CMP annealing where the microstructure is mostly stable partly due to confinement by the trench sidewalls and partly because of the previous annealing treatment (pre-CMP) the lines have already received. It is because of this reason we selected a Cu film where the microstructure had already stabilized by a previous annealing treatment.

It should be noted that there have been published reports investigating the thermomechanical response of the (1 1 1) and (1 0 0) grains measured using X-rays [28,29]. These reports demonstrate the behavior of these texture components on a macro scale but there have been no attempts made to understand the behavior of these texture components at the micro level. We report here the changes occurring in the microtexture components recorded in situ during heating of the Cu film in SEM. The novel part of the study is the investigation performed during heating of the Cu film in freestanding condition, i.e., in absence of the substrate. The information obtained from this study will directly help establish the effect of substrate, stresses and dislocations on the behavior or microtexture components. Only the results for the (1 1 1) grains have been included here as an increasing number of reports have shown the peculiar behavior of (1 1 1) grains in the Cu film [20,28]. Additionally, we have investigated two specific (1 1 1) grain orientations – $\{111\}\langle 110\rangle$ and $\{111\}\langle 112\rangle$, as these two are the commonly observed orientations in the Cu interconnects [8,30].

2. Experimental procedure

High-temperature behavior of annealed Cu film was studied in situ by EBSD in SEM under high vacuum condition of the order of 10^{-7} mbar. The Cu blanket film was electroplated to a thickness of 1 μm on a patterned thermally oxidized Si substrate presputtered by 50 nm TaN and 100 nm of Cu seed using commercial equipment. The thickness of the underlying oxide was 1 μm. The orientation of the underlying Si crystal substrate was (1 0 0)[1 1 0]. The microstructure of the film was already stabilized by annealing at 400 °C for 30 min under vacuum prior to in situ EBSD investigation. The films were subjected to two separate thermal cycles. In situ EBSD scans were performed at the same location of the Cu film at RT, 200, 400 °C and upon cooling during the first cycle. To check the reproducibility of the results in situ EBSD scans were again performed at the same location of a similar annealed Cu film during the second thermal cycle. The scans were performed at RT, 245, 425 °C and upon cooling during the second cycle. The top passivation layer was removed prior to EBSD investigation by etching in 15% HF acid for 10 min. The heating and cooling rates were on an average 7 K/min at higher temperatures but decreased to about 2–4 K/min at lower temperatures. Similar annealed Cu film was mechanically detached from the Si substrate and investigated in situ at temperatures of 255 and 450 °C starting from RT in freestanding condition. The films were roughly about 24 h at high temperatures since relatively larger areas were scanned for better statistical reliability and additionally drifting at high temperatures led to repeat scanning at some locations of the film.

The EBSD measurements were carried out in FEG SEM (Leo 1530 – Gemini) at 25 kV in square grid mode with scan step size of 75 nm in both the investigations. The working distance was 15 mm with a specimen tilt of 70°. The scanned regions varied from as small as 37×37 μm^2 to as large as 146×122 μm^2 in area for all the investigations. Computations were done using CHANNEL 5 software from HKL technology [31]. The neighboring scan data points having misorientation less than 15° were considered to be the part of a single grain. For grain size calculation the grain area was first computed from the number of scan data points comprising a grain. Assuming the computed area to be that of a circle, grain diameter was then calculated from this area equivalent circle. The grain size calculations assumed twins to be as separate grains. The minimum grain size was defined to have at least two neighboring scan steps. The minimum grain boundary misorientation angle was limited to 2°. Scanning was carried out on the film in a configuration such that the Y direction or longitudinal direction (LD) of the specimen coordinates of the Cu film was parallel to the $\langle 110\rangle$ direction of the underlying Si crystal substrate.

3. Results and discussion

Fig. 1 depicts the focused ion beam (FIB) cross-section image of the Cu film. One can see presence of columnar grain structure together with some twin lamellae. The presence of relatively large grains is the consequence of the previous annealing treatment. A 100 nm thick top passivation