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Morphological and Electrical Properties of Silver Thin Films Sputter Deposited on LTCC Substrates

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***Abstract*—Screen printed noble metal thick films are commonly used as metallization on LTCC (low temperature cofired ceramics) substrates. When, however, geometries with a lateral resolution below 20 μm are needed for the realization of devices, alternative techniques are needed, and they are provided by standard thin film technology. To minimize conduction losses, silver (Ag) is favored due to a low bulk resistivity. To evaluate the potential of Ag as metallization, thin films are sputter deposited on glass and LTCC substrates under varying conditions (i.e., plasma power) with different film thicknesses ranging up to 1.75 μm. The microstructure of the Ag films is analyzed applying techniques such as scanning electron microscopy, focused ion beam, and x-ray diffraction. With the latter approach, a mean grain size of about 33 nm is measured independent of plasma power used for Ag deposition. In contrast, the texture strongly varies with deposition parameters resulting in an enhanced generation of (111) planes at higher plasma powers due to an increased adatom mobility. Furthermore, a higher degree in (111) orientation results in a lower resistivity of the Ag films. When the Ag films are postdeposition annealed at 500°C, the resistivity decreases by a factor of 2 compared with the "as deposited" state due to grain growth. Further, sublimation and agglomeration effects dominate leading to an increase in surface roughness and resistivity above average.**

***Keywords*—Thin film technology, LTCC, Ag, resistivity, microstructure, post deposition annealing**

Introduction

Nowadays, environment detection for automotive and airborne applications based on radar sensing principles attracts much attention, due its applicability even in poor weather conditions [1]. Due to regulation aspects as well as the demand for an enhanced spatial resolution, frequency bands at 79 GHz are targeted, even for short and mid range detection regimes. Apart from dielectric losses associated with the substrate, ohmic losses in the metallization need to be reduced to a minimum to guarantee enhanced performance at the system level. Therefore, advanced metallization systems are targeted offering a high conductivity as well as a high electromigration resistance such as provided by silver (Ag) [2].

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On ceramic substrates, especially LTCC (low temperature cofired ceramics), the metallization systems which are typically used are screen printed due to the use of thick films as inner metallization layers. Due to the low temperature associated with the liquid phase sintering of LTCC, noble metal inks with low resistivity such as Au or Ag can be applied. However, the applicability of the conventional thick film printing technique is limited at high frequencies because selected components, such as antenna elements, require a lateral resolution below 10 μm. Furthermore, the resistivity of thick film conductor lines is larger compared with purely metal-based structures because of the glass-frit content and the impact of the printing process (i.e., the saw tooth effect). Although efforts are ongoing to improve the screen printing technique using photosensitive thick films, the resolution provided by thin film technology has not been achieved up to now [3, 4].

This paper focuses on the microstructure and on the electrical performance of Ag thin films on LTCC and their dependence with respect to important sputter parameters, such as the plasma power. Furthermore, the influence of postdeposition annealings up to $T_{PDA} = 800°C$ on these film properties is determined.

Experimental Details

Ag thin films are synthesized on sintered DuPont Tape 951 with a von Ardenne sputter deposition system from an 8 in target at a fixed back pressure in the deposition chamber of $P = 0.003$ mbar (Ar flow rate: 80 sccm) and varying plasma powers (i.e., $P_p = 100$ W to 1000 W). The silver target with a purity of 99.999% is fixed at a distance of 65 mm from the substrate. After evacuation of the deposition chamber below 5×10^{-6} mbar, Ar is introduced to presputter the target for 2 min prior to each film deposition for purification purposes. Titanium (deposition parameters: $P = 0.003$ mbar, Ar flow rate: 80 sccm, $P_p = 300$ W) with a thickness of about 10 nm is used as adhesion promoter for the Ag top metallization.

In all depositions the substrates are nominally unheated and the Ag film thickness is 1 μm except otherwise stated. The postdeposition annealings (PDA) up to 800°C are done in a Nabertherm muffle furnace (Nabertherm, Lilienthal, Germany) under atmospheric conditions. For the fabrication of test devices, a mixture of ammonium hydroxide (NH_4OH) (32%) and hydrogen peroxide (H_2O_2) (35%) is used as the wet chemical etchant for the Ag thin films.

The x-ray diffraction (XRD) technique was applied for crystallographic analyses performing θ-2θ scans in the Bregg-

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Brentano mode. The equipment (Philips X-Pert (Panalytical, Almelo, The Netherlands)) works at a wavelength of 1.5418 Å corresponding to the CuKα-line. From the XRD measurements the mean grain size and the microstrain is deduced according to the Williamson-Hall method, described in detail elsewhere [5].

The maximum roughness depth R_{max} and the average roughness R_a of LTCC substrates and Ag thin films are determined with a profilometer (Alphastep 200) from Tencor Instruments (Milpitas, Ca, USA) at a fixed scan length of 2 mm. In order to monitor the microstructure of the Ag films at different annealing levels, analyses with a scanning electron microscope (SEM) in a Dual Beam FIB-FEI Strata DB235 (Hillsboro, OR, USA) workstation are performed which also allows in situ cross-sectioning.

Electrical characterization is performed with a digital multimeter Prema 6001 (Mainz, Germany) in the two-point measurement configuration. To determine the film resistivity conductor lines having three different lengths are realized. Plotting the total resistance versus the geometry factor of each test structure, the resistivity can be gained from the sloop when the film thickness is known. Doing so, the impact of parasitic effects arising, for example, from the wiring is reduced to a minimum.

Results and Discussion

Pre-investigation on the deposition rate and the wet chemical etch rate are done on conventional glass substrates to keep the measurement error as low as possible due to the low surface roughness of glass (R_a < 1 nm) compared to LTCC ($R_a \approx$ 700 nm) [6]. As the amount of kinetic energy of the ions impinging on the substrate is expected to have a strong influence on the texture formation in sputter deposited silver thin films on silicon [7], the plasma power P_P is varied from 100 W to 1000 W assuming a similar behavior on LTCC. Fig. 1 shows a series of XRD spectra of Ag films sputter-deposited with various plasma powers on the glass-ceramic substrate. Independent of sputter parameters these data indicate a face centered cubic (fcc) crystal lattice when compared with data of the corresponding powder diffraction file (PDF) for fcc Ag (PDF number 04-0783).

In accordance with results published for Ag thin films on glass substrates [8], the ratio of planes in (111) compared with the (200) direction also increases on LTCC substrates with higher plasma power levels (see Fig. 2). When keeping the pressure in the deposition chamber constant, Ag particles impinging on the substrate gain a higher kinetic energy, thus resulting in enhanced adatom mobility during film growth. Therefore, the energetically most favored plane as the (111) having the lowest surface energy [15] is predominantly generated when compared with lower plasma power levels.

The influence of texture on the electrical properties is measured on glass substrates exemplary for samples sputter-deposited at P_P = 100 W and P_P = 1000 W. As reported in Birkholz et al. [9], the resistivity is decreased even up to temperatures of 300°C for those films yielding a higher degree of (111) orientation (see Fig. 3).

The TCR can be determined from the slope of these characteristics to $\alpha_f = 3.15 \times 10^{-3}$ K^{-1} (P_P = 100 W) and $\alpha_f = 3.57 \times 10^{-3}$ K^{-1} (1000 W), respectively. These values are reduced when compared with the data reported in the literature for Ag bulk material (i.e., $\alpha_b = 4.1 \times 10^{-3}$ K^{-1}) [10]. This decrease in the TCR of film compared with bulk specimens is basically attributed to a lower mean grain size, thus counteracting the increase in resistivity originating from pure phonon scattering due to an increasing portion of the current which is temperature activated across the grain boundaries [11].

According to the Williamson-Hall approach [5], the mean grain size d_g can be deduced from the XRD data. Up to P_P = 900 W, a value of about 33 nm (±2 nm) is deduced indepen-

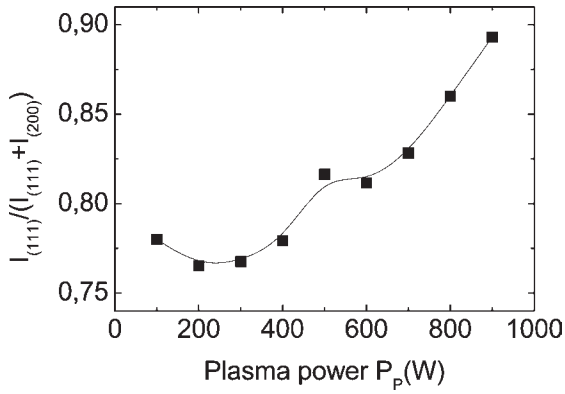


Fig. 2. Increasing ratio of (111) to (200) planes in the Ag thin films at higher plasma powers P_P.

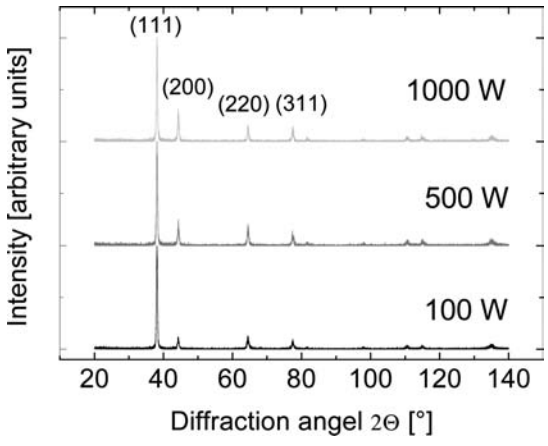


Fig. 1. XRD spectra of 1 μm thick Ag thin films sputter-deposited on LTCC.

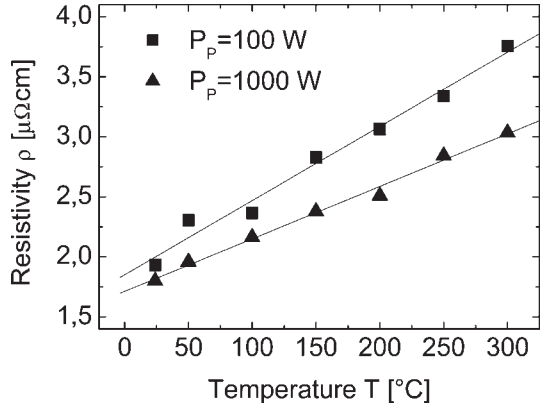


Fig. 3. Resistivity ρ as a function of temperature T of Ag films deposited on glass substrates at different plasma powers.

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dent of plasma power. This value is in good agreement with Sugawara et al. [12] reporting a mean grain size of about 30 nm in Ag thin films deposited on oxidized silicon substrates. At the 1000 W sample, however, a slightly coarser graining with d_g = 40 nm is determined which is again attributed to the enhanced adatom mobility during film growth. In contrast to the parameter P_p, the film thickness has a higher impact on the mean grain size, as illustrated in Fig. 4.

During deposition, the grain growth starts at nucleation sites that are statistically distributed on the rough and inhomogeneous LTCC surface. Due to a low substrate temperature of the nominal unheated substrate close to room temperature, shadow effects play an important role at this early stage of film coalescence, especially at low film thicknesses, resulting in an increasing grain size with d_f. The local minimum at d_f = 1.25 µm is attributed to the strong increase in microstrain and hence, in film stress (see Fig. 5), indicated by similar curve characteristics. The high film stress generates defects such as stacking faults in the film [13], which may serve as nucleation sites for the growth of additional grains. Having passed this characteristic film thickness, a further increase in d_f reveals a decrease in microstrain at a simultaneous increase in d_g.

Due to the targeted implementation into a standard LTCC process flow, which requires high refiring temperatures, Ag thin films were temperature loaded in air up to annealing temperatures (T_{PDA}) of 800°C. From cross-sectional views of 1 µm thick Ag thin films on LTCC prepared by the FIB technique, a fine grained film structure is observed in the "as deposited" state up to T_{PDA} = 130°C (1 h), as shown in Fig. 6(a), supporting the results gained from the XRD measurements on glass substrates. Fig. 6(b) yields the coarse-grained microstructure of

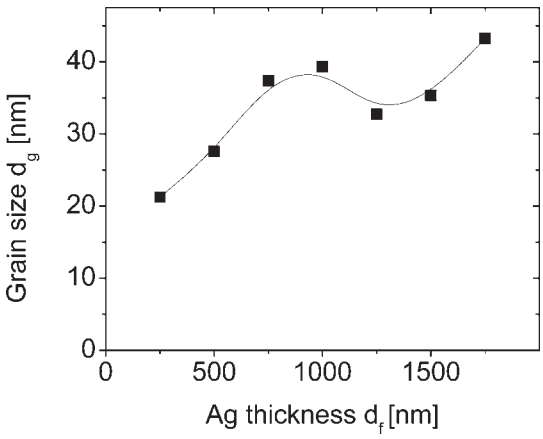


Fig. 4. Increasing grain size d_g at larger Ag film thickness d_f. The Ag films are deposited at P_P= 1000 W.

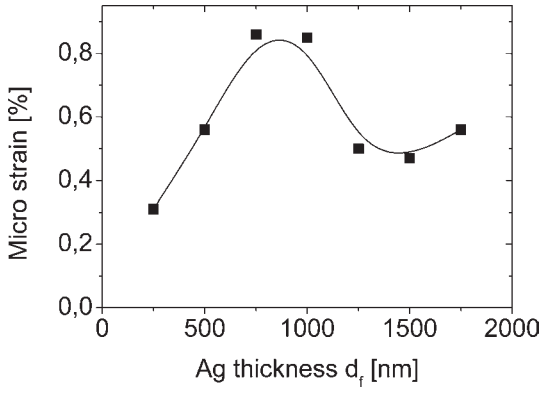


Fig. 5. Increasing microstrain as a function of Ag film thickness.

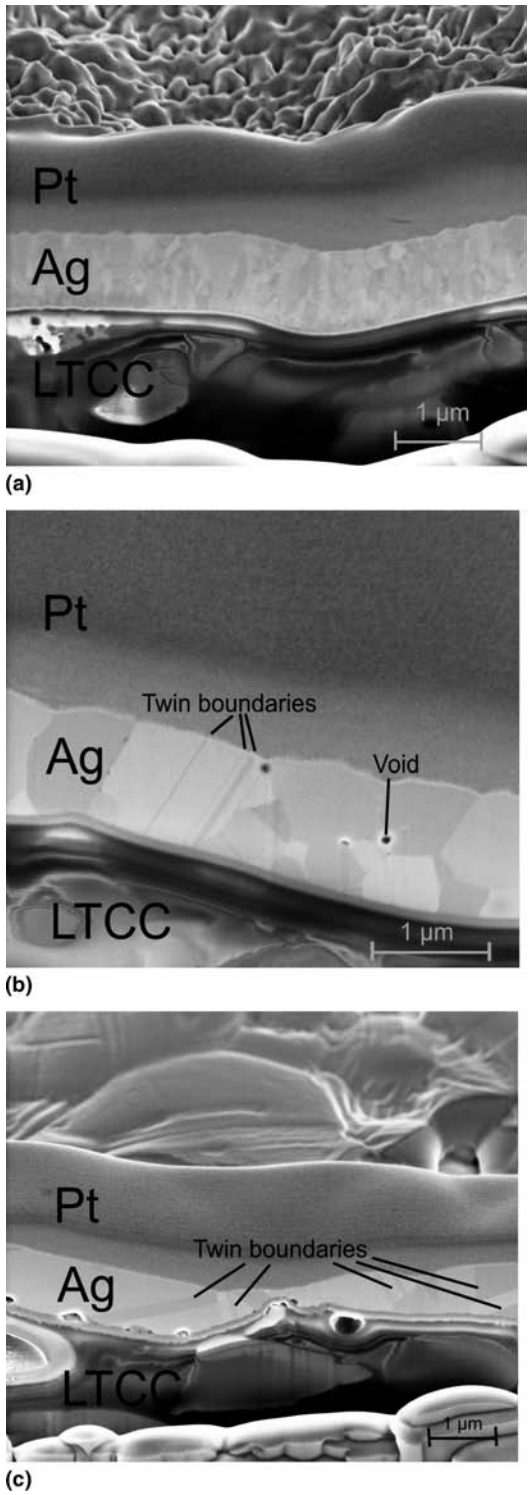


Fig. 6. Cross-sectional view of Ag films after annealing at (a) T_{PDA} = 130°C, (b) T_{PDA} = 400°C, and (c) T_{PDA} = 800°C.

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an Ag film annealed at 400°C. Apart from the growth in grain size, defects such as voids and twins are generated. They originate from the high film stress which again arises from the large mismatch of the temperature coefficients of expansion associated with the Ag films (bulk value: $\gamma = 19.6 \times 10^{-6}\ K^{-1}$) compared with the LTCC substrate ($\gamma = 5.8 \times 10^{-6}\ K^{-1}$ for DuPont Tape 951 (Wilmington, DE, USA)). Upon annealing at 800°C for 1 h, these effects are activated even further and a high number of twin dislocations are generated in the film, as illustrated in Fig. 6(c). From the cross-sectional investigations, it is clearly observed that the overall layer thickness is locally reduced and very inhomogeneous due to sublimation and agglomeration effects.

To evaluate the impact of thermal loading on the electrical performance, the resistivity is measured at room temperature upon annealings up to 800°C.

As larger grains are generated during the annealing process, the resistivity decreases from $\rho = 3.3\ \mu\Omega\cdot$cm in the "as deposited" state to a minimum value of about 1.8 μΩ·cm upon exposure at $T_{PDA} = 500$°C (see Fig. 7), comparable to the bulk value of 1.6 μΩ·cm [14].

The higher resistivity of Ag on LTCC, similar to the value on glass (i.e., $\rho = 1.8\ \mu\Omega\cdot$cm) in the "as deposited" state can be understood by the enhanced roughness of the glass-ceramic substrate. The nucleation behavior, however, is regarded to be similar as LTCC has a glassy surface coverage. This assumption is verified by comparable values for d_g, as illustrated in Fig. 8(a), yielding a mean grain size of about 30–40 nm, also determined from AFM measurements [6].

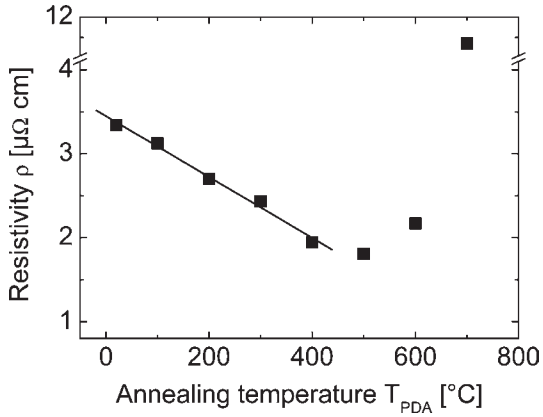


Fig. 7. Resistivity of Ag thin films on LTCC vs. annealing temperature.

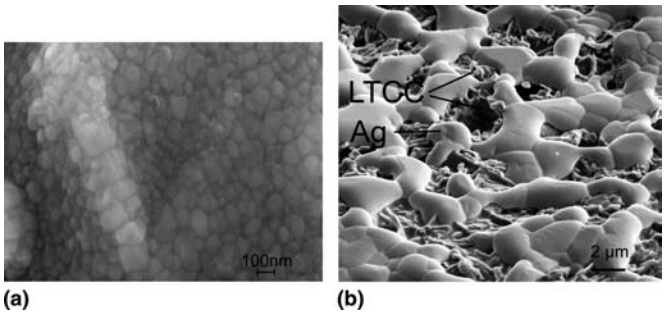


Fig. 8. (a) Surface morphology of a 1 μm thick Ag thin film on LTCC. (b) Surface morphology of a 1 μm thick Ag thin film after temperature loading at $T_{PDA} = 800$°C.

Above $T_{PDA} = 500$°C, the increase in ρ is caused by a decreasing film thickness compared with the original value [see Fig. 6(a) and 6(c)]. In this annealing range, the onset of effects such as sublimation and even agglomeration dominate the electrical performance. Starting from thermal induced microgrooves evolving prominently at triple-points, islands are formed at $T_{PDA} = 800$°C which are connected to each other by ridges, thus indicating the completeness of the agglomeration process [see Fig. 8(b)]. Due to the discontinuous nature of the Ag film, the resistivity increases above average. Upon annealing at 800°C the film degrades so strongly that no ohmic behavior is measured.

Conclusions and Outlook

In this study, Ti/Ag thin films sputter-deposited on conventional LTCC substrates are investigated. From XRD measurements it is deduced that the probability of (111) plane formation is enhanced when applying higher plasma power levels due to a higher adatom mobility of the impinging Ag particles. Furthermore, a correlation is found between the texture and the electrical conductivity of the Ag thin film deposited on glass substrates. Basically, the resistivity decreases with a higher degree of (111) orientation. Secondly, the plasma power has only low impact on the microstrain and on the mean grain size which is about 33 nm. In contrast, both quantities are strongly increasing with film thickness up to a characteristic value of about 1.25 μm. Above that value, it is assumed that the generation of crystallographic defects, such as stacking faults, acting as additional nucleation sites influence the graining predominantly. Annealing the Ag thin films up to 500°C leads to a decrease in resistivity by a factor of 2 compared with the "as deposited" state. Loading the sample with $T_{PDA} > 500$°C, sublimation and even agglomeration effects dominate, resulting in an increase in resistivity above average when assuming the original film thickness.

In general, it has to be stated that there is a lack of compatibility with conventional thick film technology as postfiring temperatures range up to 850°C. Despite this limitation in thermal budget, further research will focus on the influence of post deposition annealings on film texture. Furthermore, the long time reliability will be analyzed by electromigration tests applying accelerated aging conditions.

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