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Research paper

Dynamics of filling process of through silicon via under the ultrasonic agitation on the electroplating solution

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ABSTRACT

Through-silicon-via (TSV) connection is a promising technology to provide more effective and faster data processing in integrated-circuit (IC) devices. In this study, via-filling processes with the direct ultrasonic agitation, the effects of additives (accelerator, suppressor and leveler) and different current densities are investigated. Microvias with a diameter of 20 μm and a depth of 65 μm is used in the electrodeposition process. The dynamic evolution of the via-filling process activated by ultrasonic under different current densities is obtained by scanning electron microscopy. It is found that the application of ultrasonic agitation can change the deposition rate of copper ion at the via and improve the filling process to obtain void-free TSVs. Under the action of ultrasonic agitation, the via-filling process under the condition of low, middle and high current density are explored to realize the "V" type filling, "U" type filling, void filling, respectively. In addition, the filling ratio and filling speed of copper in the vias under different conditions are also analyzed in this work.

1. Introduction

In the next generation of IC packaging technique, three-dimensional (3D) integration has become a major focus for more effective and faster data processing in integrated-circuit (IC) devices. Three-dimensional integration is mainly achieved by interconnecting vertically stacked chips using through-silicon-via (TSV). In this process, Cu is considered as an excellent material due to its high electrical conductivity and cost-effectiveness. Nowadays, copper electroplating is widely practiced and plays an important role in the process of filling the microvias. Nevertheless, problems still exist in this approach, of which the most critical one is the existence of seams and voids during the TSVs filling process.

To avoid the possible reliability problem or unstable performance at high frequency caused by such micro defects, the via-filling process to fabricate void-free TSV is investigated [1–6]. It is known that superfilling (bottom-up filling) is an accepted filling performance to fulfill this purpose. To realize the superfilling, a common chemical method is to add additives into the plating bath to modulate the performance of the copper electrodeposition in vias. The additives such as suppressor, accelerator, and leveler, which are absorbed on the electroplating surface, are used to change the current density distribution in the vias to achieve the bottom-up filling [7–11]. In addition to adding additives, physical methods are also adopted in superfilling, such as using pulsed current [12–14], applying ultrasonic field to improve the filling process. The ultrasonic agitation improves mass transport [15] inside vias and decrease the concentration gradient of reactants within via to avoid the formation of voids or seams. Chen et al. investigated the copper electroplating with the assistance of ultrasonic agitation and found that, ultrasonic agitation has the potential to facilitate the formation of void-free TSV with a high aspect ratio [16]. Moreover, the fabrication of the nanostructures assisted by the ultrasound is studied by Phuruangrat [17]. So far, there are few reports about the effect of ultrasonic agitation combining the effects of additives on the filling process of TSV.

In this work, a novelty method for the TSV filling process with the direct ultrasonic agitation on the electroplating solution with additives is explored. With the same current density and plating conditions, the application of ultrasonic agitation can change the deposition rate of copper ion in the via, improve the TSV filling ratio, and realize bottom-up filling. Under the action of ultrasonic agitation, the dynamic via-filling process under the different current densities is explored. The “V” filling could be achieved at low current density (0.002 A/cm^2), and the “U” (conformal) filling can be achieved at middle current density (0.005 A/cm^2). At high current density (0.008 A/cm^2), the via is pinched off and a large void is formed.

2. Experimental

In this experiment, a small piece of silicon which is cut off from a silicon wafer containing micro-vias is used as a cathode fixed on the electrochemical platinum electrode clamp (Xuzhou Zhenghao Electronic

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Technology Co. Ltd.). The internal surface of the silicon wafer and the blind via is covered with a thin copper layer. The piece size is 1 cm in width, 2 cm in length. The diameter of the via on the piece is 20 μm and 65 μm in depth. A planar copper containing phosphorus with an area of 33 cm^2 is as an anode immersed in electroplating solution. The precision power (Agilent B2901A) is used to supply a direct current. The ultrasonic horn of the ultrasonic processor (Shang Chao Pai) is used to apply ultrasonic wave propagated by longitudinal motion to the plating bath. As shown in Fig. 1, the plating bath used in the experiment is Haring cell with 25 cm in length, 8.5 cm in width, 12.5 cm in height and 0.5 cm in thickness. The anode is fixed on one side of the plating bath, while the cathode clamp is fixed in the center of the bath and the distance between them is 5 cm. The distance between the cathode and the upper surface of solution is 1 cm. The ultrasonic horn through the middle bracket is fixed on the rod between the cathode and the anode. The ultrasonic horn's bottom is immersed in the solution with 4 cm in depth (The volume of plating solution is 1 L). In this experiment, the composition of copper electroplating solution contains: 195 g/L $CuSO_4 \cdot 5H_2O$ (Sinopharm Chemical, China), 49 g/L H_2SO_4 (Sinopharm Chemical, China), 0.05 g/L NaCl (Sinopharm Chemical, China), 0.01 g/L MPS (Mengde, China) as accelerator, 1.5 g/L PEG (Mw. 6000, Mengde, China) as suppressor, and 0.02 g/L PN (Mengde, China) as leveler. The reagents of the plating solution maintain the optimal concentration combination according to our previous experiments. The solvent is deionized water and the experiment is carried out at room temperature.

It is worth noting that before the experiment starts, the silicon wafer should be pretreated as the following process. Firstly, electrode holders are used to clamp the silicon piece containing blind vias and it is all immersed into deionized water in the beaker. Secondly, the silicon piece containing blind vias is put into a vacuum device for pumping vacuum processing. When the vacuum degree reaches −15 kg/cm^2, the condition is maintained for 5 min. At this point, bubbles are observed on the silicon wafer. Thirdly, an ultrasonic cleaning machine is applied to clean the silicon wafer for 5 s by immersing it in the deionized water to make the wafer surface free of obvious bubbles. The second and third steps are repeated for 3 to 4 times, until no bubbles can be found on the surface of the silicon wafer. At the beginning of the experiment, the silicon wafer with vias (cathode) is soaked in the electroplating solution for 15 min. The solute is diffused into the vias, which makes the solute concentration be relatively balanced in and out of the via. After the silicon wafer is fully soaked, the precision power and ultrasonic processor are switched on to supply power and ultrasonic wave, and the time is recorded. In this experiment, the current densities are set to 0.002 A/cm^2, 0.005 A/cm^2, and 0.008 A/cm^2; the ultrasonic power is set to 60 W; the frequency is set to 20 kHz. For different current densities, the corresponding electroplating time is also different. When the current density is 0.002 A/cm^2, the plating time is 0.5 h, 1 h, 1.5 h, 2.5 h, and 5 h, respectively. When the current density is 0.005 A/cm^2, the plating time is 15 min, 30 min, 45 min, 60 min, and 90 min, respectively. When the current density is 0.008 A/cm^2, the plating time is 10 min, 20 min, 30 min, 45 min, and 60 min, respectively. When the plating is completed, the silicon chip is sealed out by resin. Its surface is then cleaned with water and blown to dry immediately. After the solidification of the resin, a lapping and polishing machine is used to polish samples to obtain the maximum cross-section of vias. Then, the samples are immersed in alcohol and cleaned with the ultrasonic cleaning machine until no copper debris exists in the hole. Finally, SEM pictures of the samples are taken for further observation and analysis.

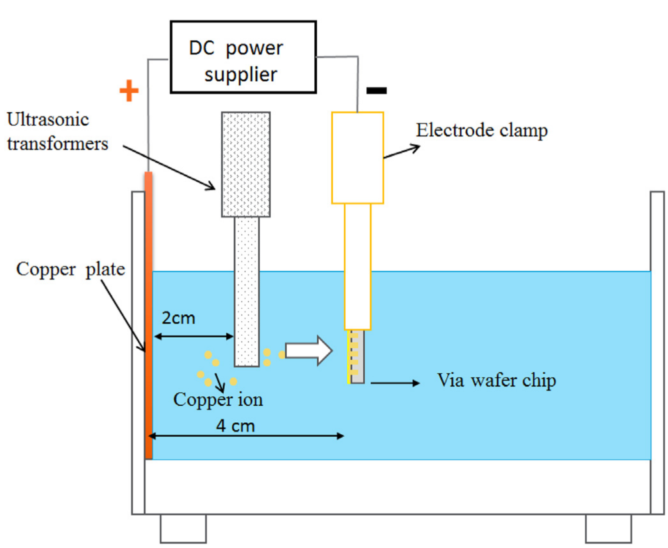

Fig. 1. Electroplating system with ultrasonic agitation.

After electron microscope image is obtained through SEM, the filling ratio is calculated by image processing technology. MATLAB is used to process the obtained data. In order to describe and analyze the experimental results accurately, the filling ratio and the filling growth ratio are defined as follows:

Filling ratio

$$M_i = B/(B_i + S_i) \tag{1}$$

Filling growth ratio

$$V_i = (M_i - M_{(i-1)})/(T_i - T_{(i-1)}) \quad i \gg 1 \tag{2}$$

where S_i is the unfilled area in vias and B_i is the filled area in vias as shown in Fig. 2.

3. Results and discussions

3.1. Comparing of TSV filling with and without ultrasonic

When no ultrasonic wave is applied during the filling process, the current density is set to 0.002 A/cm^2, 0.005 A/cm^2, 0.008 A/cm^2 respectively and the plating time is 5 h. The profiles of the filled TSVs using different current densities are shown in Fig. 3. When the current density is

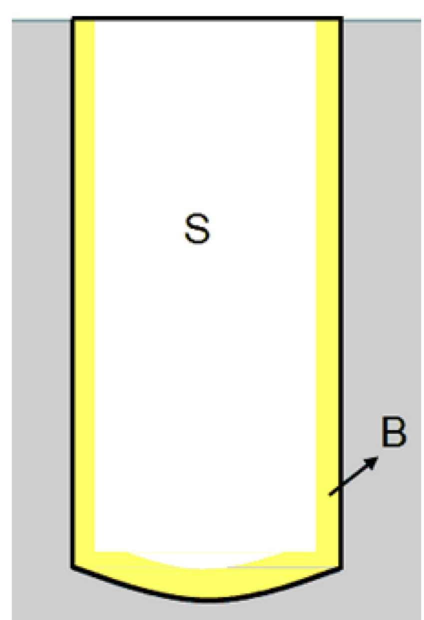

Fig. 2. Definition of the filled area "B" and the unfilled area "S" in cross-section of TSV filled by Cu.

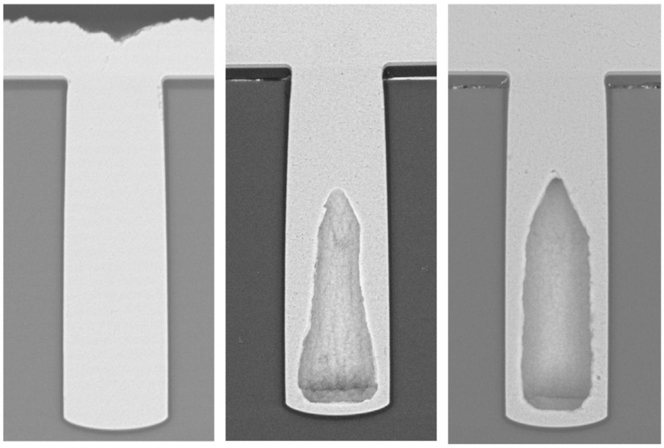

Fig. 3. Blind-via electroplating without ultrasound in 5 h.

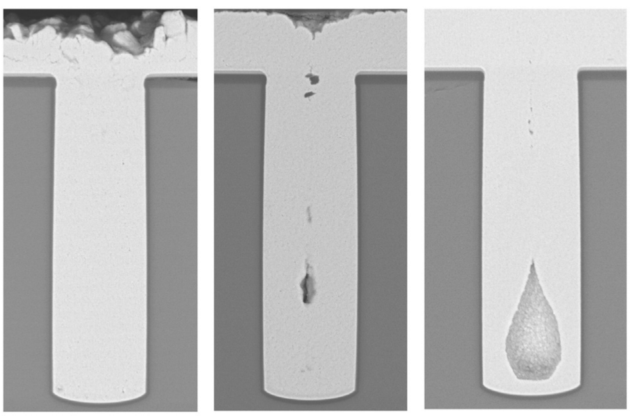

Fig. 4. Blind-via electroplating with ultrasound in 5 h.

0.002 A/cm^2, TSV filling ratio M_i can reach 100%, i.e. no seams and voids in the hole. However, when the current density is increased to 0.005 A/cm^2, a huge void is formed in the hole and the filling ratio is 75.62%. When the current density is further increased to 0.008 A/cm^2, the filling ratio decreases to only 62.38%. When the ultrasound is applied to the electroplating process, the profiles of the filled TSVs using different current densities (0.002 A/cm^2, 0.005 A/cm^2 and 0.008 A/cm^2) after 5 h plating are shown in Fig. 4. When the current density is 0.002 A/cm^2, the filling ratio can reach 100%. When the current density is 0.005 A/cm^2, a narrow seam forms in the middle part of the TSV hole and the filling ratio reaches 98.76%. When the current density is increased to 0.008 A/cm^2, a void can be observed in the TSV hole and the filling ratio is 87.11%. The formation of voids and seams can be ascribed to the undesirable distribution of current density and insufficient mass transfer in vias. Obviously, the filling ratio with ultrasonic is increase 23.14% with current density of 0.002 A/cm^2 and 24.73% with current density of 0.005 A/cm^2, respectively. It is clear that the application of ultrasound can significantly improve the mass transfer in the TSVs and increase the filling ratio. In order to find out the mechanisms of such favorable effects, the TSV dynamic filling process is further explored under the electroplating condition with ultrasonic wave of the current density 0.002 A/cm^2, 0.005 A/cm^2 and 0.008 A/cm^2 respectively.

3.2. Dynamic TSV filling process with ultrasonic

The TSV filling process at distinct plating time (0.5 h, 1 h, 1.5 h, 2.5 h, and 5 h) under the electroplating condition with ultrasonic wave and the current density of 0.002 A/cm^2 is shown in Fig. 5. It can be seen that in the early stage of electroplating, the filling speed of bottom is faster than the opening part of the via, which makes the thickness of the deposited Cu on the bottom increase rapidly. The opening part is suppressed by the suppressor, which causes the copper layer to grow slowly. At the time of 0.5 h, the filling ratio is 25.05%. Since then, the thickness of the deposited Cu on the bottom increases rapidly, and the filling ratio reaches 45.60% at 1 h. When the plating is carried out to 1.5 h, the section of the filling part of the hole presents "V shape" visibly, and the filling ratio is 60.37%. At 2.5 h of the filling process, the most area of the via is filled (the filling ratio is 85.08%) and the unfilled part still maintains the "V shape". It is clear that in the entire process of filling, the copper deposition of the opening of the via is suppressed, which makes the deposition speed at the bottom of the via faster than that at the opening of the via. Therefore, the TSV can achieve bottom-up filling. No voids and seams are produced in the TSV with a 100% filling ratio with current density of 0.002 A/cm^2.

The filling results as shown in Fig. 6 at the plating time of 15 min, 30 min, 45 min, 60 min and 90 min under the condition with ultrasonic and the current density of 0.005 A/cm^2. At the electroplating time of 15 min, the growth thickness of copper at the via bottom is greater than that at the opening. The filling profile shows a "U" shape and the filling ratio is 28.11%. When plating time comes to 30 min, the thickness of the deposited copper at the bottom is increased further and the filling ratio is 48.12%. When the plating time is from 30 to 45 min, the growth speed of the deposited copper accelerates, and the filling ratio is 66.45% at 45 min. At 60 min, the thickness of the deposited copper at the opening is almost identical to that at the bottom of the via and the filling ratio is 80.66%. When the plating time comes to 90 min, the via is filled completely. To sum up, at the beginning of filling process, the deposition speed of the bottom is larger than the opening part. However, along with the via-filling process, the suppressor at the opening decreases, caused by the change of the via shape and the interaction of the additives. Thus, the deposition rate of copper increases at the opening, which results in an identical deposition rate at the bottom and at the

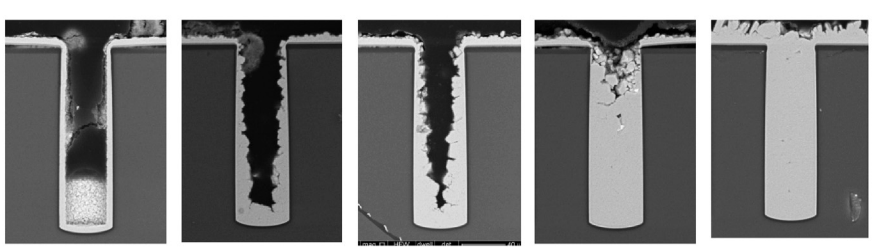

Fig. 5. Morphology at distinct plating time under ultrasonic field with current density of 0.002 A/cm^2.

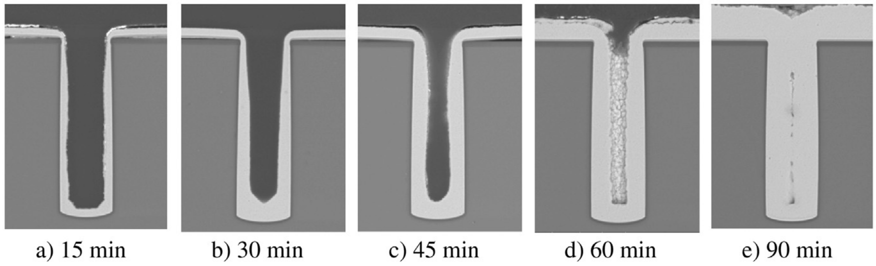

Fig. 6. Morphology at distinct plating time under ultrasonic field with current density of 0.005 A/cm^2.

opening of the via. Finally, a narrow seam forms at the middle of the via and the filling ratio reaches to 97.16%. Compared to electroplating with the current density of 0.002 A/cm^2, the time required to fill the via is reduced to only 90 min.

The results of filling at distinct electroplating time (10 min, 20 min, 30 min, 45 min and 60 min) under the plating condition with ultrasonic and the current density of 0.008 A/cm^2 is shown in Fig. 7. In the first 10 min of the plating, the thickness of the deposited copper of the via is consistent, and the filling ratio is 29.96%. From 10 to 20 min, the growth rate of the deposited thickness at the opening is still slow, but the thickness at the bottom and middle grows rapidly and the filling ratio comes to 48.49% at 20 min. Then the growth rate of the deposited thickness at the via's center is accelerated. There is a tendency of closure at the via's center and the filling ratio of 30 min is 63.46%. When electroplating is carried out at 45 min, the middle part of the via is closed, and the filling ratio is 79.47%. When the plating time comes to 90 min, the opening of the via is fully filled. Obviously, in beginning stage of the electroplating, TSV is filled with 'U' shape. During the filling process, the suppress effect on the middle becomes weakened, which results in the filling rate of copper at the middle of the via accelerating significantly. Due to the narrow part of the hole, the cupric ions cannot be transferred to the bottom of via timely. Thus the deposition rate of copper at the bottom further slows down and an empty hole is formed. Thereafter, the cavity is retained in TSV. When the current density is 0.008 A/cm^2, a larger void is left at the bottom of the via (the filling ratio is 85.21%) and the filling time is further shortened.

As we know, the filling mode is the decisive factor for the filling quality. As shown in Fig. 3, ultrasonic agitate can improve the TSV filling quality. To compare the electroplating process under three different current densities, it is obvious that by varying the current density under the action of ultrasound, different filling modes are realized. According to the experimental results of Yang et al. [18] and Zhu et al. [19], the current density and the concentration of the additives have an important influence on the filling mode. Ultrasound can produce cavitation effect in the solution [15,20], which can cause the forced convection of the solution, and enhance the mass transfer process. Therefore, the exchange rate of various substances in the solution is accelerated, and the various ions consumed in the via can be supplied timely, so that the filling mode can be improved.

3.3. Analysis of filling ratio and filling speed in dynamic filling process

The filling ratios with different current densities during the filling process are shown in Fig. 8. The curves of the filling ratios of the three levels of current density show a rising trend with the increasing of plating time, which is normalized in the figure. The three filling ratio curves show a gentle rise and the curvature decreases gradually with decreasing the current density. The gaps and voids exist in the TSV filling with current density of 0.005 A/cm^2 and 0.008 A/cm^2, and the filling ratio can only reach 96.37% and 85.21% ultimately.

The change of filling speed versus normalize plating time is shown in Fig. 9. The filling speeds of the three current densities show a decreasing trend with the increase of electroplating time. A possible explanation is that the changes of the via morphology lead to the decrease of the interface between copper ions in the solution and the contact part, resulting in a decline of the deposition speed of copper. In addition, for electroplating time from 45 to 60 min with 0.008 A/cm^2, the fill speed is low due to the presence of voids. Filling speeds show a trend of rapid decline, during the plating time of 60 to 90 min with 0.002 A/cm^2, 30 to 45 min with 0.005 A/cm^2 and 20 to 30 min with 0.008 A/cm^2, respectively, which is mainly due to the great changes of the morphology of the vias.

4. Conclusions

In this paper, the dynamics of electrodeposition process of TSV with ultrasonic using different current densities is studied. It is concluded that with the same current density and plating conditions, the application of ultrasonic agitation can change the deposition rate of copper ion at the via and improve the filling process to obtain void-free TSVs. Under the action of ultrasonic wave, low current density (0.002 A/cm^2) could result in the "V" (bottom-up) filling with 100% filling ratio, i.e. no seams and voids. With the current density of 0.005 A/cm^2, "U" (conformal) filling can be obtained, and a narrow seam exists in this condition. With the current density of 0.008 A/cm^2, a large void is formed near the bottom of the via. Under the action of ultrasound, using different current densities on the TSV-filling process can realize different filling mode. In addition, it is also found that in the process of plating, the filling speed decreases with the increase of plating

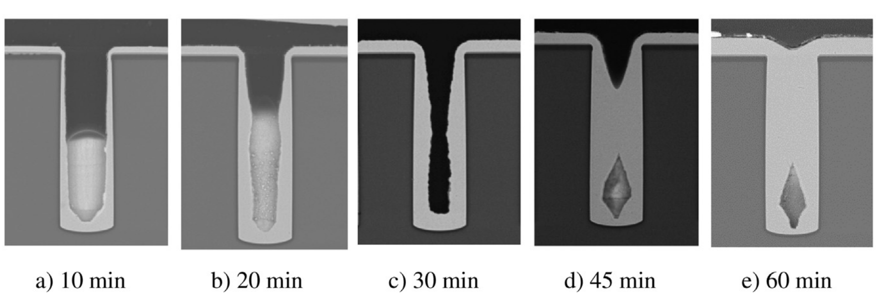

Fig. 7. Morphology at distinct plating time under ultrasonic field with current density of 0.008 A/cm^2.

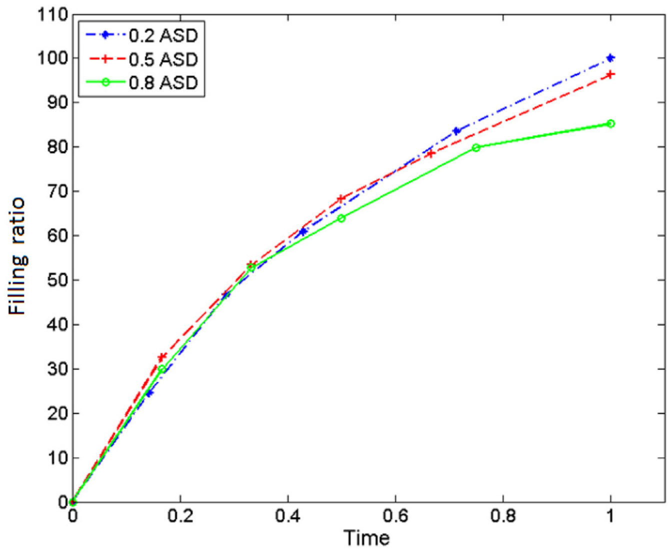

Fig. 8. The filling ratios with different current densities (0.002 A/cm^2, 0.005 A/cm^2, and 0.008 A/cm^2) along normalize time.

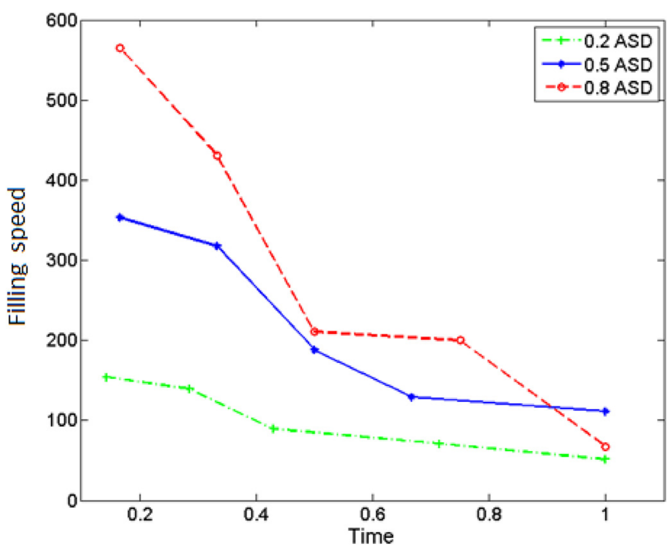

Fig. 9. The filling speeds (um^3/min) with different current densities (0.002 A/cm^2, 0.005 A/cm^2, and 0.008 A/cm^2) along normalize time.

time, rather than keeping a constant value. This paper focus on the influence of ultrasound with fixed power and frequency on the morphology of the via filling. In next step, the influence of the parameters of ultrasound such as power and frequency, the cavitation effect on the copper electrodepostion and behaviors of the additives need further explored.

Acknowledgments

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