

Investigation of Microstructure of Pressure-assisted Ag Sintering Layer by Deep-etching Method

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Abstract: Nano silver (Ag) film was used as the bonding material. High quality bonding layers with low porosity (1.2% ~1.4%) were obtained when applying sintering pressure of 5~10 MPa at 250 °C. The microstructure of the sintered layers was investigated by deep-etching method in comparison with the bulk Ag. The results reveal that the deep-etched morphology of the sintered layer is quite different from that of the bulk Ag. Micron-scale polyhedral Ag grains are observed in the deep-etched morphology of the sintered layers. The grain size and the area of the interfaces between these polyhedral Ag grains change as the sintering pressure increases from 5 MPa to 10 MPa, which is considered as the dominate factor affecting the bonding properties of the sintered layers. As the sintering pressure increases from 5 MPa to 10 MPa, the shear strength of the pressure-assisted sintering specimens is improved.

Key words: bonding; reliability; sintering; microstructure; deep-etching

The third-generation semiconductor materials such as SiC and GaN lead to the fast development of power electronics^[1-4], and bring a higher requirement on the bonding technology of power chips^[5,6]. Due to reliability concerns, traditional bonding methods, such as lead-free soldering and adhesive bonding, are not suitable for high power and high temperature applications^[7-9]. Because of the ability of serving at high temperature, silver (Ag) sintering is considered as a promising method to the assembly of power chips^[10-12]. Since Ag sintering is a non-melting bonding process, the bonding performances are highly suppressed by the pores in the layer^[13]. Researchers tried to improve the sintering quality by adding extra forces such as electrical current, ultrasonic, and mechanical forces, among which pressure-assisted Ag sintering is considered as an effective bonding method. Yu et al^[14] reported that the shear strength of the Ag sintering layer is enhanced by increasing the sin-

tering pressure during the sintering process. However, the mechanism causing the bonding enhancement by increasing sintering pressure is still not clear. Therefore, deep-etching technique was used in this study to investigate the microstructure of the sintering layer and to discuss the effect of sintering pressure on the microstructure and the shear strength of pressure-assisted Ag sintering bonding.

1 Experiment

The sintering material was a nano Ag film with the thickness of 65 μm (provided by ALPHA). The optical morphology of the Ag films used in this study is shown in Fig.1. The high accuracy sintering equipment (Sinterstar) was provided by Boschman Technologies B.V. In order to investigate the morphological transformation of the nano Ag particles, the Ag film was heated at 250 °C for 3 min. The morphology and the constituent of the Ag film before

Received date: January 17, 2019

Foundation item: National Natural Science Foundation of China (51604090); Natural Science Foundation of Heilongjiang Province (E2017050); University Nursing Program for Young Scholars with Creative Talents in Heilongjiang Province (UNPYSCT-2015042)

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and after pressure-less sintering were characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD), respectively. To investigate the effect of sintering pressure on the microstructure and shear strength, dummy dies (9.4 mm×9.4 mm×1.2 mm) were sintered onto the substrates (13.6 mm×13.6 mm×2 mm) at 250 °C for 3 min under the sintering pressure of 5 and 10 MPa. Here the dummy dies and the substrates were molybdenum (Mo) plates with 5 μm Ag-coated layers on their surfaces. Cross-sectional morphology and constituent of the bonding layers were studied by SEM and energy dispersive spectrometer (EDS), respectively. To investigate the morphology of the microstructure, bonding layers as well as bulk Ag were deep-etched using an etching solution (1% HCL+1% HNO₃+98% CH₃COOH). By this method, the specimen was etched very deeply at the interfacial areas between the Ag grains. The obtained three-dimensional morphology is helpful for the better understanding of the sintering mechanism in this study. The shear strength of the specimens was evaluated by the Instron 5569 mechanical tester at the shear speed of 0.3 mm/min.

2 Results and Discussion

The SEM micrographs of the nano Ag particles on the films can be observed in Fig.2a and 2b. As shown in the figure, the Ag film shows a nest-like microstructure. Here the grain sizes of the nano Ag particles are 50~200 nm and these nano particles are connected dendritically. XRD patterns in Fig.3 indicate that the corresponding crystal plane index of the film is (111), (200), (220), (311) and (222). In

order to investigate the morphological transformation of the nano Ag particles, the Ag film was heated at 250 °C for 3 min. As shown in Fig.3, the heating process has a limited effect on the XRD patterns. Meanwhile, as presented in Fig.2c and 2d, the morphology of the heated film is similar to the original one shown in Fig.2a and 2b. Compared with the microstructures in Fig.2b and 2d, the grain sizes of the nano Ag particles in the film are larger after the sintering process. Therefore, the pressure-less sintering process has a limited effect on the morphology and the constituent of the Ag film, but leads to the growth of the nano Ag particles.

Fig.4a shows the cross-sectional SEM micrograph of the pressure-assisted sintered specimen under 5 MPa at 250 °C for 3 min. Based on the results of EDS spectra in Fig.4b and 4d, the constituent of each layer in Fig.4a is clear. Here the top and the bottom layers are dummy die and substrate Mo, respectively. The dark layer next to the Mo layer is the Ni

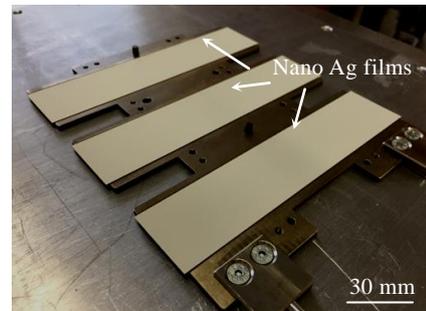


Fig.1 Nano Ag films on the Mo substrates

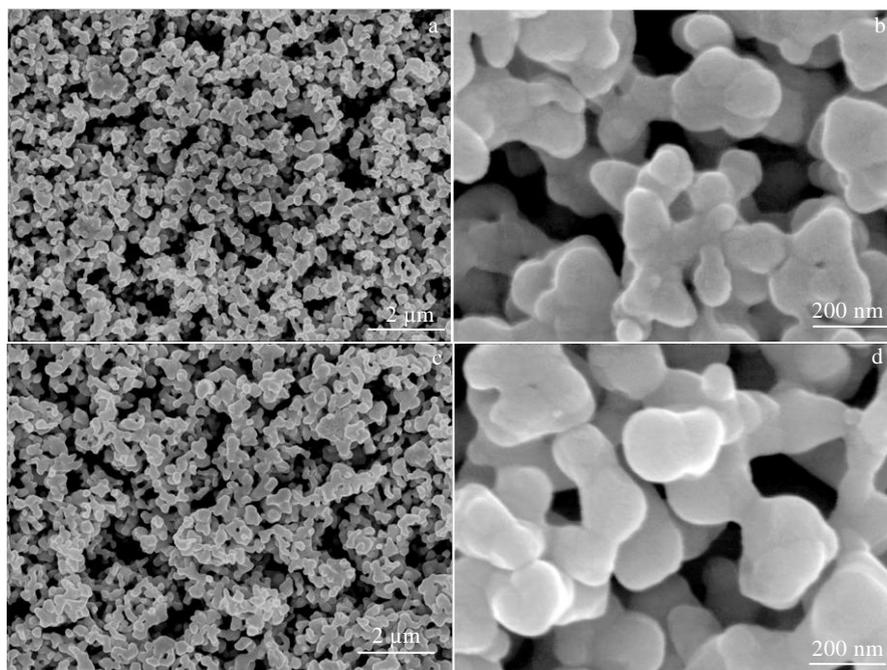


Fig.2 SEM images of nano Ag film before (a, b) and after (c, d) pressure-less sintering at 250 °C for 3 min

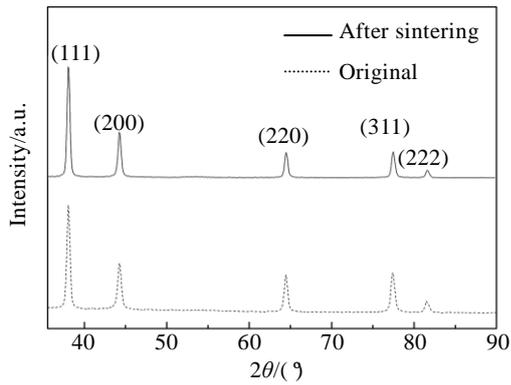


Fig.3 XRD patterns of the films before and after pressure-less sintering

and Cu coated layers on the Mo plates. Here the total thickness of the Ni and Cu layers is about 2 μm . The layer next to the Cu layer is the Ag-coated layer with the thickness of 5 μm at the surface of the Mo plate. The Ag sintered layer can be observed at the central area in Fig.4a. According to the magnified micrograph of the sintered Ag layer, as shown in Fig.4c, the pressure-assisted sintered layer shows a much more condensed microstructure than the pressure-less sintered layer in Fig.2c. It is obvious that sintering pressure plays an important role in the bonding and densification of the nano Ag film.

Fig.5a shows the deep-etched morphology of the Ag layers sintered under 5 MPa. Compared to Fig.4a, the etched specimen shows a much more different morphology because the Ag at the interfacial areas is etched and removed

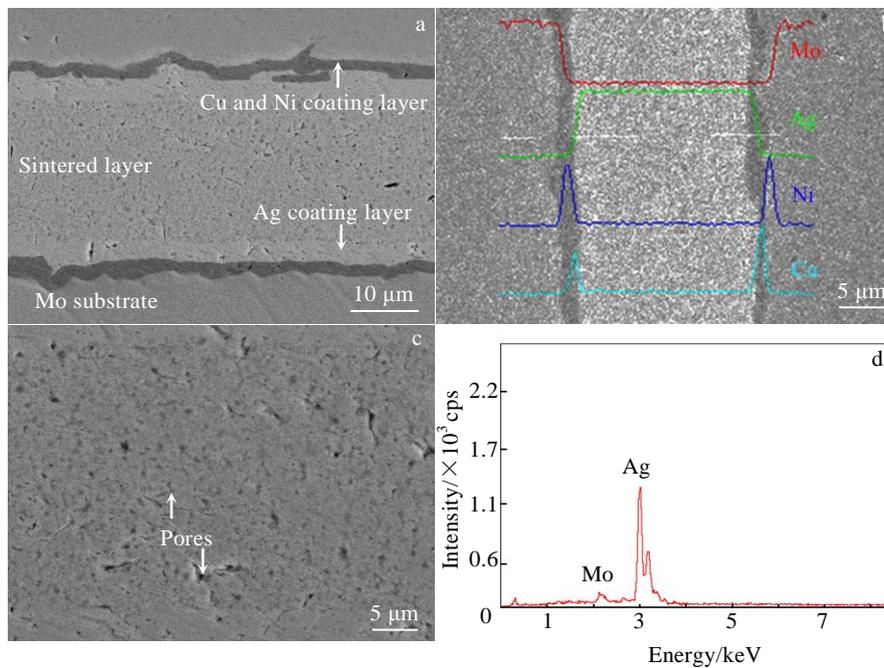


Fig.4 Cross-sectional SEM morphologies and EDS analysis of the pressure-assisted sintering specimen: (a) SEM image, (b) EDS line-scan result, (c) magnified sintered Ag layer, and (d) EDS spectrum of the sintered Ag layer

during the etching process. The magnified micrograph of the etched sintered layer is shown in Fig.5b. As shown in the figure, polyhedron Ag grains can be observed. Here the average size of the polyhedron grains is about 1.7 μm . And these grains show hollow appearances. According to the morphology of the sintered layer without deep-etching in Fig.4c, the holes inside the polyhedron grains are suggested to be the etched areas instead of the pores formed after the sintering process. Fig.5c and 5d show the deep-etched morphology of the sintered Ag layers under 10 MPa. Polyhedron Ag grains also appear in the etched micrograph. However, the average grain size of the micron-scale polyhedron Ag grains in the specimen under the sintering pressure of 10 MPa is smaller than under 5 MPa. Further mag-

nified micrograph, as shown in Fig.5e, reveals that these micron-scale polyhedron Ag grains are formed by nano Ag grains with the size of 50 nm approximately.

Literatures have reported that increasing sintering pressure promotes the densification of Ag-sintered layers^[15, 16]. As shown in Fig.5a and 5c, the sintered Ag layer under 10 MPa exhibits lower thickness than under 5 MPa. Therefore, the polyhedron Ag grains under higher sintering pressure are condensed and show smaller grain size. Similar phenomenon has been obtained by Shao et al^[17], whose study showed that increasing sintering pressure leads to a decrease in the grain size of nano ZnO sintered layers.

In order to analyze the microstructure of the sintered Ag layers, bulk Ag was also deep-etched in comparison. The

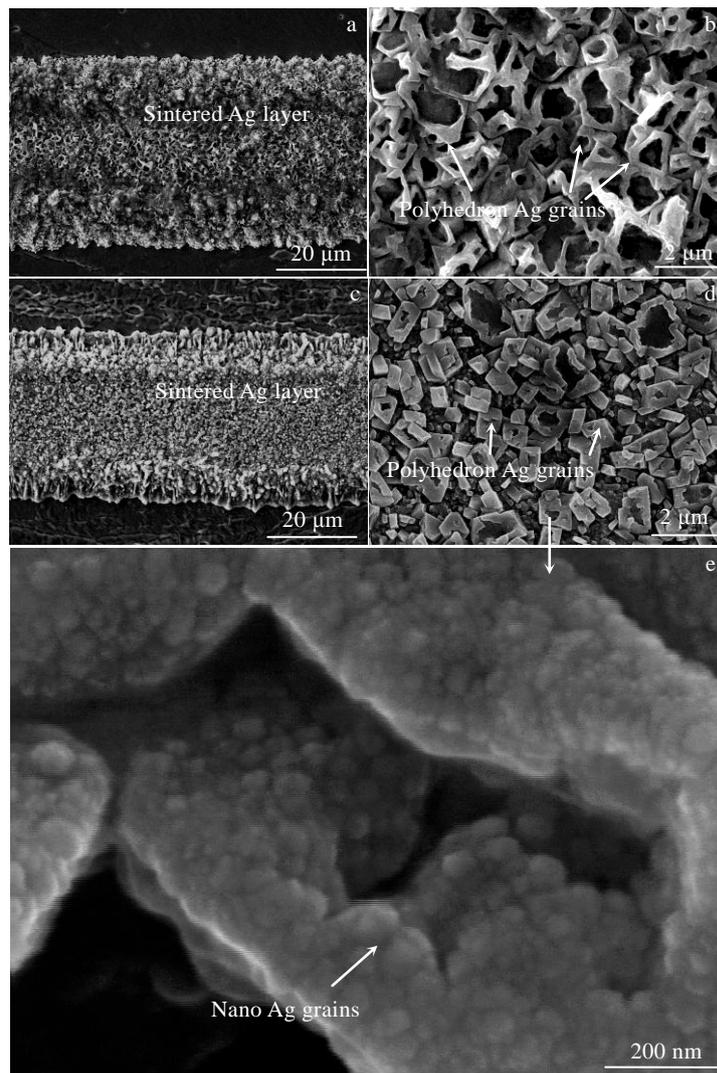


Fig.5 Deep-etched morphology of the Ag layers sintered under 5 MPa (a, b) and 10 MPa (c~e)

morphology of the etched bulk Ag is shown in Fig.6. It is clear that the deep-etched morphology of the bulk Ag is quite different from that of the sintered Ag layers. As shown in Fig.6a, the microstructure of the bulk Ag is composed of the Ag grains which are less than 1 μm . The magnified view in Fig.6b indicates that these grains are formed of nano Ag grains. Compared with the deep-etched microstructure in Fig.5, it is obvious that the microstructure of the sintered layer is different from that of the bulk Ag. This is because the forming mechanism of the sintered Ag layer differs from that of the bulk Ag. The sintered Ag layer is obtained mainly due to the deformation and the solid diffusion of the nano Ag particles. Higher sintering pressure provides higher energy for the deformation and the diffusion of the nano Ag particles, thus forming smaller polyhedron Ag grains. However, as shown in Fig.5 and Fig.6, the sintered layer does not have a microstructure as fine as the bulk Ag. This is considered as a reason why the comprehensive properties

of the sintered layer are not as good as that of the bulk Ag.

Fig.7 shows the thickness, porosity, grain size, and the shear strength of the pressure-assisted sintered Ag layers under 5 and 10 MPa. As the sintering pressure increases from 5 MPa to 10 MPa, the thickness of the sintered layer decreases from 20.3 μm to 19 μm . Meanwhile, the porosity in the sintered layer decreases from 1.39% to 1.29%. Although the porosity of the sintered layers has little change due to the increase of the sintering pressure, the shear strength of the sintering specimen is significantly enhanced from 44.19 MPa to 69.41 MPa, as shown in Fig.7d.

The morphologies of the shear fractures are shown in Fig.8. As presented in Fig.8a and 8c, the shear fractures appear at both the Ag sintered layer and the sintered interface between Ag and coating layer. Meanwhile, the fractures transfer from the Ag sintered layer to the sintered interface due to the increase of sintering pressure. It means

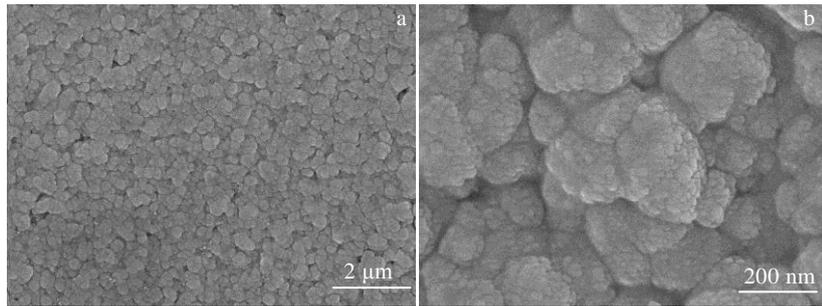


Fig.6 Deep-etched morphology of the bulk Ag

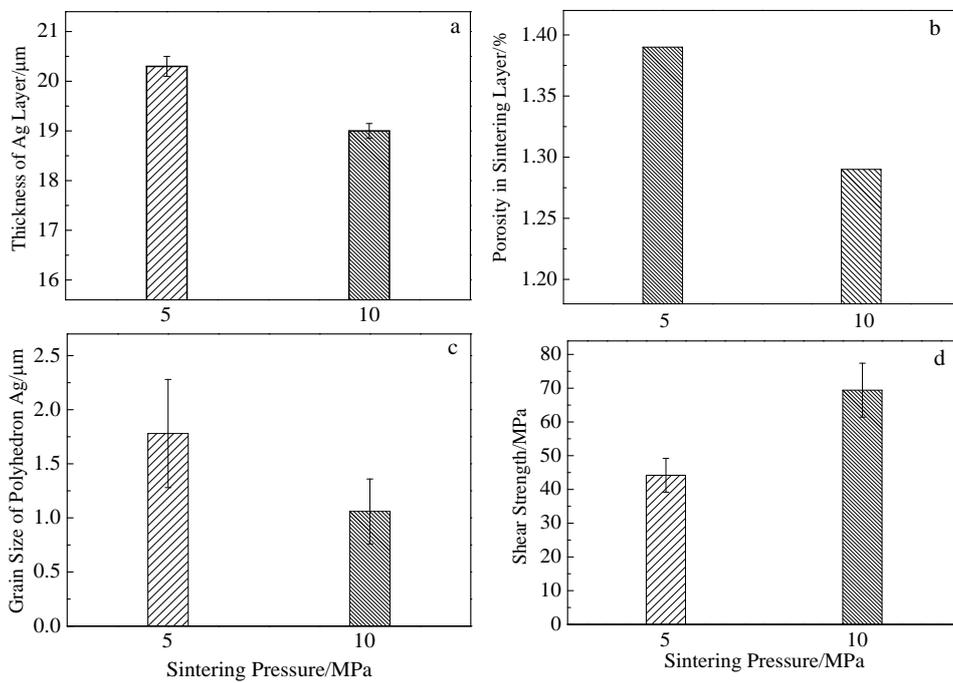


Fig.7 Thickness (a), porosity (b), grain size (c), and shear strength (d) of the pressure-assisted sintering Ag layers under 5 and 10 MPa

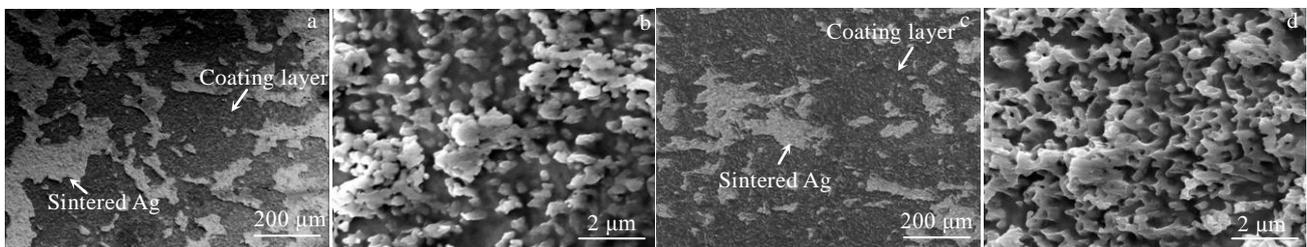


Fig.8 SEM images of the shear fractures sintered under 5 MPa (a, b) and 10 MPa (c, d)

that the strength of the Ag sintered layer is enhanced with the increase of the sintering pressure from 5 MPa to 10 MPa. Fig.8b and 8d show the magnified morphologies of the fracture in the Ag sintered layers. Clear Ag grains on the

shear fracture can be observed, as shown in Fig. 8b. In contrast, the shear fracture under 10 MPa shows ductile morphology in Fig.8d. According to the results and discussion of the deep-etched microstructure, the increase of the sin-

tering pressure promotes the refinement of the polyhedron Ag grains. The average grain size of the micron-scale polyhedron Ag grains decreases from 1.78 μm to 1.06 μm . The refinement and densification of the polyhedron Ag grains are considered as the dominant factor improving the bonding strength.

3 Conclusions

1) High quality Ag sintered layers with low porosity (1.2% to 1.4%) can be obtained using a nano Ag film under the sintering pressure from 5 MPa to 10 MPa.

2) Micron-scale polyhedron Ag grains are observed in the sintered Ag layers by a deep-etching method. The interfaces between these polyhedron Ag grains are the key factor affecting the properties of the low porosity sintered layers.

3) By applying higher sintering pressure, the size of the polyhedron Ag grains decreases and the area of the interfaces between these polyhedron Ag grains increases. This is considered as the dominate reason for the improvement of the bonding strength of the sintered layers.

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基于深腐蚀技术的纳米银压力烧结接头微观组织研究

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摘要: 采用纳米银薄膜作为研究对象, 在 250 $^{\circ}\text{C}$ 下施加 5~10 MPa 的烧结压力, 获得低孔隙率(1.2%~1.4%)的高质量烧结接头。随着烧结压力由 5 MPa 增大至 10 MPa, 接头抗剪切强度显著提升。借助深腐蚀技术对烧结接头及块体银的微观组织结构进行对比分析。结果表明, 烧结接头的深腐蚀形貌与块体银存在明显差异。烧结接头在深腐蚀条件下呈现大量微米尺度的多边形银晶粒, 当烧结压力由 5 MPa 增大至 10 MPa 时, 多边形晶粒的尺寸与晶粒间的连接面积发生明显变化。这一现象是影响纳米银压力烧结连接接头性能的主控因素。

关键词: 连接; 可靠性; 烧结; 微观组织; 深腐蚀

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