

# OPTIMIZATION OF PRE-TREATMENT CONDITIONS FOR ELECTROLESS DEPOSITION ON SMALL PADS FOR UBM FORMATION

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## Abstract

The plating process of under bump metallization (UBM) formation is important to add solder joint and wire bonding properties for the aluminum alloy pads on the silicon wafer. We studied the process where the electroless Ni can be deposited uniformly on sputtered or vapor deposited aluminum pads in various crystal orientations. Because damage to the aluminum often occurs during the pretreatment process, we also studied the pretreatment process with the goal of minimizing aluminum damage.

## Introduction

Electroless plating for UBM formation technology on the silicon wafer has garnered attention due to its advantages of low-cost and high productivity. The ENIG (Electroless Nickel / Immersion Gold) and ENEPIG (Electroless Nickel / Electroless Palladium / Immersion Gold) processes are of particular interest because they are suitable for solder bumping. Furthermore, ENEPIG and ENAG (Electroless Nickel / Electroless Gold) are also recognized for excellent wire bonding (W/B) characteristics, and these processes are expected to become more common in the future. UBM is routinely formed on wafer pads sputtered with aluminum in these processes. On the other hand, electroless plating is a wet process, therefore the pretreatment process is an important aspect of UBM formation.

Generally, a zincate coating is applied to an aluminum electrode treated after a conditioner, etch and acid rinse. The zincate film is typically deposited from highly alkaline zincate solutions. Electroless nickel plating commences after the zincate. However, the size and orientation of aluminum crystal can be altered by deposition conditions. It is possible that the zincate film will not deposit normally on the aluminum surface. In this paper, we focus on the optimized pretreatment process for aluminum termination pads with special attention on etching and zincating on polycrystalline aluminum. Furthermore, we considered the mechanism of electroless nickel deposition for these materials. Additionally, zincate processes often cause partial corrosion (“spikes”) in the aluminum coating because the zincate solution is highly alkaline. We investigated the relationship between zincate components and spikes in the aluminum, and the relationship

between zincate components and adhesion between aluminum and nickel.

## Experimental

### A) Aluminum orientation and etching

We used aluminum-silicon sputtered films (silicon=1.0%) on silicon substrate for this test. Test wafers were prepared with two film conditions which differed in grain size: small (<1.0um) and large (>100um). The electroless plating process for aluminum-silicon material is shown in Table 1. Each chemical was selected from our commercial product line. Two types of etching (etching A and etching B) were used on two kinds of prepared wafers. Etchant A was a typical alkali type; etchant B contained added silver. Electroless nickel was plated to about 1um in order to observe the uniformity of coverage. DI water rinsing was applied between process steps and samples were air-dried. The surface condition in each process was observed by FE-SEM (Ultra55, Carl Zeiss). Cross-section observations were made using FIB (SMI2050, Hitachi High-Tech) to evaluate the electroless Ni deposit uniformity.

Table 1. The electroless plating process for Al-Si material

Test wafer	Grain size (small)	Grain size (large)
Conditioner	Commercial product	
Etching	Etching A (Commercial product)	Etching B (Etching A with silver)
Acid rinse	Nitric acid	
1 <sup>st</sup> zincate	Commercial product	
Acid rinse	Nitric acid	
2 <sup>nd</sup> zincate	Commercial product	
Electroless nickel	Commercial product	

## B) The spike-less plating process for Al material

Blanket wafers or TEG wafers, available commercially, were used. Both types of wafers were aluminum-copper sputtered (copper=0.5%) on the silicon substrate. The TEG wafer has specified pad defined by silicon nitride passivation. The electroless plating process for aluminum-copper material is shown in Table 2. Each chemical was selected from our commercial product inventory. Three types of zincate solutions were prepared for this test. Zinc concentrations of each solution were fixed based on the commercial product and only iron concentrations differed. The zincate deposit was produced under the same conditions (temperature and immersion time) using these zincate solutions. DI water rinsing was used after each process step; samples were air-dried. The surface condition of the zincate deposit was observed by FE-SEM (Ultra55, Carl Zeiss). The cross-section was observed to evaluate the aluminum spike using FIB (SMI2050, Hitachi High-Tech). The zinc and iron depositions in the zincate film were determined by the film dissolving method using an atomic absorption spectrometer (Z-5300, Hitachi High-Tech). Field emission type Auger electron spectroscopic-analysis equipment (JAMP-9500F, JEOL, measure range: 100 x 100  $\mu\text{m}$ ) was used to analyze the elemental depth profile in the zincate deposit. The adhesion between aluminum and nickel was examined using the nickel bump shear test method, the nickel bump was sheared by a shearing tool (170 $\mu\text{m}/\text{sec}$ , Dage series #4000) after 15 $\mu\text{m}$  of nickel plating on 100 $\mu\text{m}$  square pads from the TEG wafer, and the shear strength was compared.

Table 2. The electroless plating process for Al-Cu material

Process	Chemical
Conditioner	Commercial product
Acid rinse	Nitric acid
1 <sup>st</sup> zincate	Commercial product (Various Fe concentration)
Acid rinse	Nitric acid
2 <sup>nd</sup> zincate	Commercial product (Various Fe concentration)
Electroless nickel	Commercial product

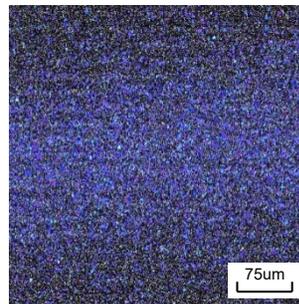
## Results and Discussion

### A) Aluminum orientation and etching

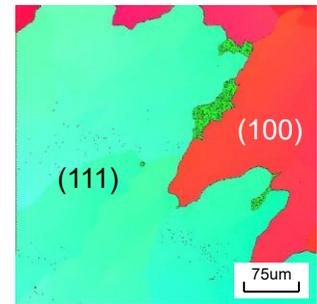
Fig.1-A and Fig.1-B show the EBSD analysis (OIM system, TSL measure range: 400 x 400  $\mu\text{m}$ ) of the aluminum-silicon test wafer used for the experiments. Fig.1-B is a measurement of aluminum surface of large grain size. The examined crystal orientation is shown, such as (111) and (100) planes, and the grain size was greater than 100 $\mu\text{m}$ . On the other hand, as Fig.1-A shows, the small grain size aluminum surface constituted from poly-crystalline deposit and was mostly oriented in the (111) plane. Moreover, the grain size was less

than 1 $\mu\text{m}$ .

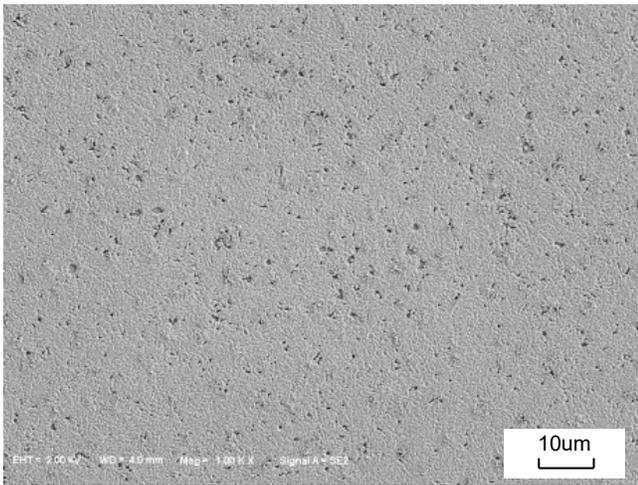
The FE-SEM images after the second zincate film are shown in Fig.2-A ~ 2-c. The zincate was deposited over almost all areas by treating with etching solution A when using a small grain size wafer. However, the zincate deposit was incomplete when using the large grain size wafer. We conclude that the difference of aluminum orientation causes this phenomenon. On the other hand, a uniform zincate film was deposited on the entire surface regardless of the orientation when using etching solution B. The FE-SEM image after electroless nickel plating is shown in Fig.3-A ~ 3-C. These results indicate that the nickel deposition is dependent on the surface condition after the second zincate film. A precise zincate film is needed to deposit nickel uniformly. The cross-section SIM image after electroless nickel plating when using a large grain size wafer and different etching solutions is shown in Fig. 4. The measurement points of cross section observation were located on the boundary line of the orientation side of the sputtered aluminum. By these SIM images, the differences in the aluminum orientation are evident. The wafer pretreated by etching solution A exhibited nickel skip deposition at the boundary line of the orientation side. Moreover it was observed that the aluminum was etched superficially in the nickel skip point. On the other hand, when the wafer was pretreated by etching solution B, nickel deposited uniformly, not related to the aluminum orientation. This was confirmed by evaluation of the cross-section. The same degree of aluminum etching was observed according to the difference of the aluminum orientation plane. This proves that etching is key to achieving uniform nickel deposition. A properly deposited zincate with the appropriate etching chemical leads to uniform electroless nickel deposition.



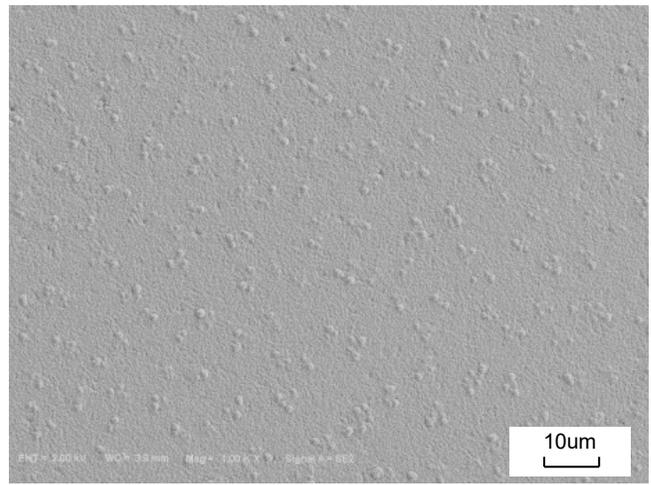
(Fig.1-A) EBSD image of a test wafer (small grain size)



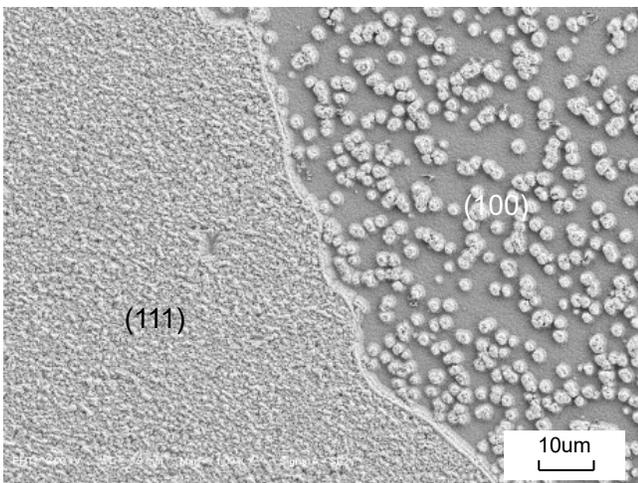
(Fig.1-B) EBSD image of a test wafer (large grain size)



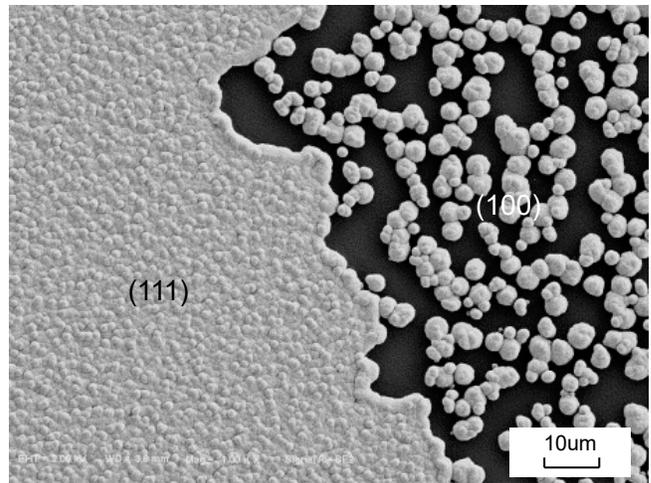
(Fig.2-A) FE-SEM image after secondary zincate film (Small grain size, etching A)



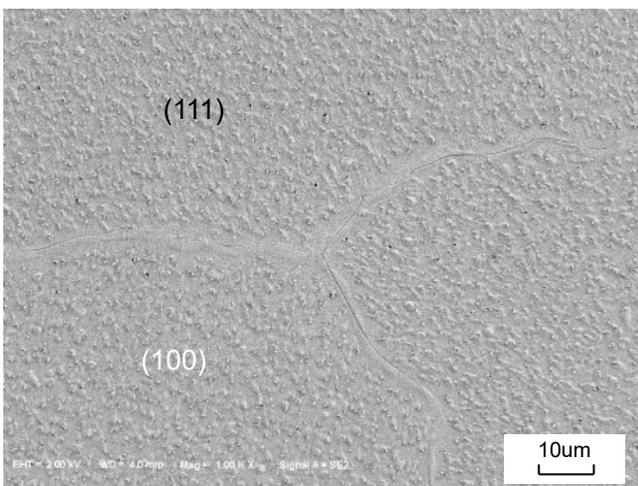
(Fig.3-A) FE-SEM image after electroless Ni plating film (Small grain size, etching A)



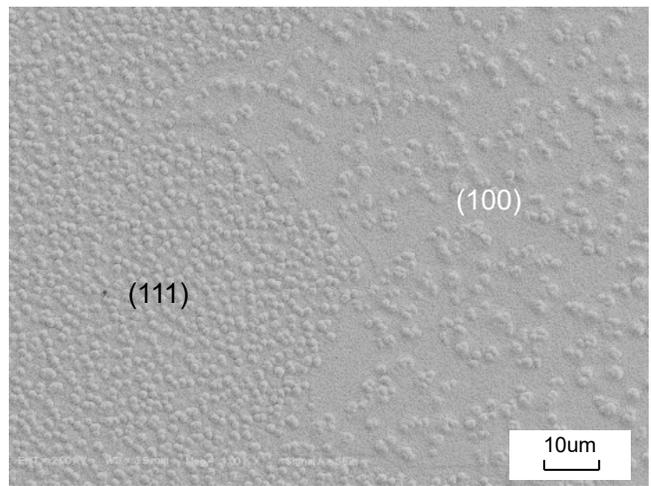
(Fig.2-B) FE-SEM image after secondary zincate film (Large grain size, etching A)



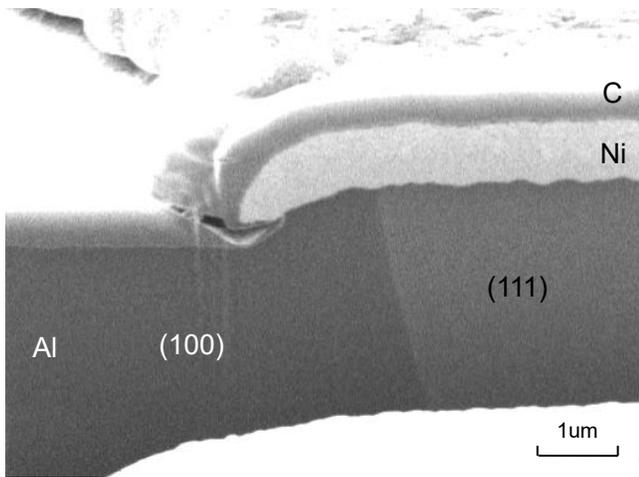
(Fig.3-B) FE-SEM image after electroless Ni plating film (Large grain size, etching A)



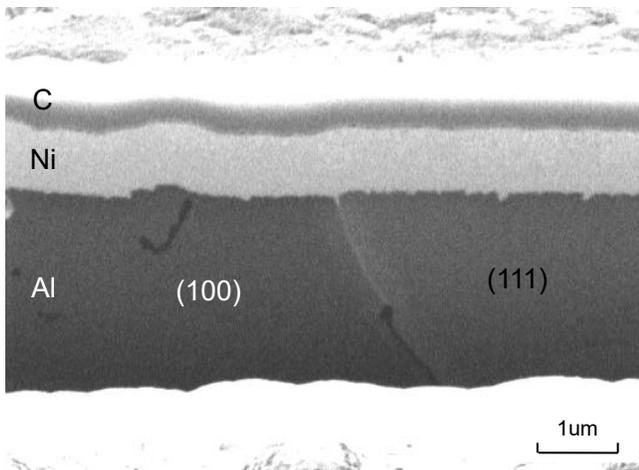
(Fig.2-C) FE-SEM image after secondary zincate film (Large grain size, etching B)



(Fig.3-C) FE-SEM image after electroless Ni plating film (Large grain size, etching B)

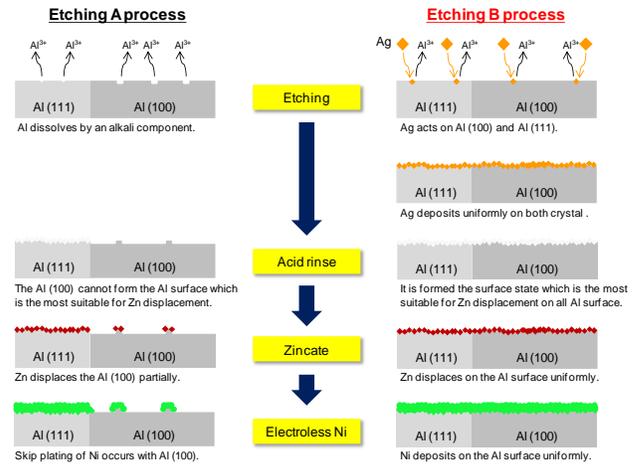


(Fig.4-A) Cross section SIM image after electroless nickel plating (Large grain size, etching A)



(Fig.4-B) Cross section SIM image after electroless nickel plating (Large grain size, etching B)

We postulate the mechanism of this process shown in Fig.5. By displacement reaction of aluminum and zinc, the zincate treatment entirely covers the aluminum surface with dense particles of zinc, and this forms the starting point of nickel plating reaction with the zinc. Regarding zinc deposition in the zincate treatment, it is known that displacement will advance in the convex part on the aluminum surface.<sup>1)</sup> The surface after etching the aluminum (100) side is flatter compared with other orientation planes, and there are few convex areas used as the deposit starting point of zinc in the zincate treatment. However, regardless of orientation, the deposit of silver takes place because the silver potential is more noble than aluminum, like the etching solution B. When silver deposits, the dissolution of aluminum takes place and forms a uniform convex shape in the aluminum surface. We presume that the convex part becomes the starting point of the deposition of the zinc film. These phenomena are expected to be similar with the small grain size wafer. However, since the grain size is small, the area of the aluminum (100) plane where zincate film doesn't deposit is also small. Therefore, it is believed that aluminum is completely covered by electroless nickel plating.

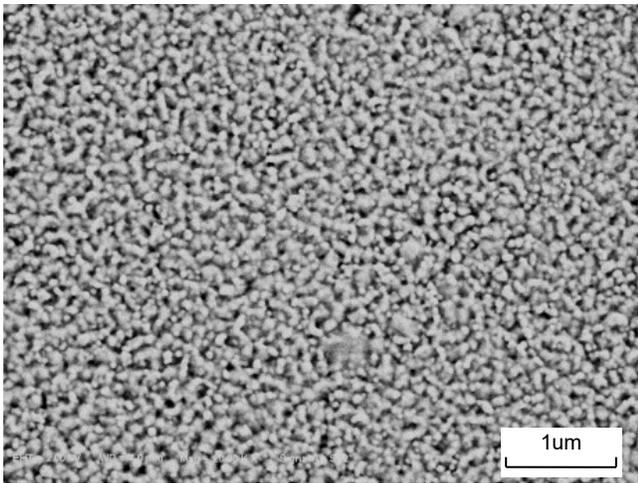


(Fig.5) Reaction mechanism by different etching

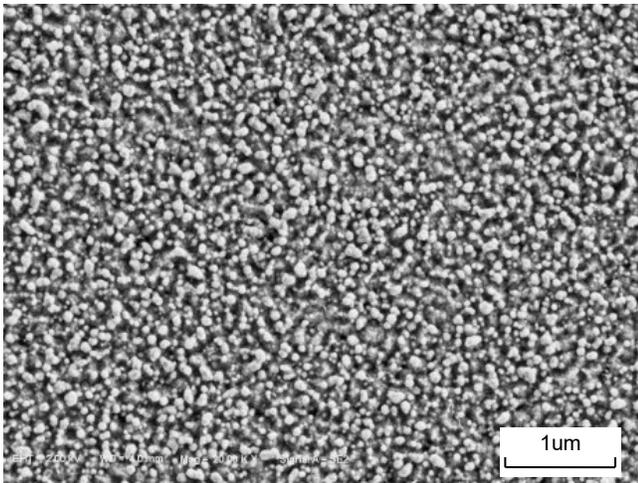
### B) The spike-less plating process for aluminum material

Generally, iron is contained in the zincate solution to improve adhesion between aluminum and nickel. In order to check for aluminum spikes and adhesion, testing was performed using our commercial product and fluctuating the iron concentration. The FE-SEM image after the second zincate film is shown in Fig. 6-A ~ 6-C, and the result of the displacement amount of zinc and iron in the zincate film is shown in Fig.7. As mentioned, the zinc concentration in the zincate solution was fixed, and only the iron concentration was changed. The iron concentration of our commercial product was labeled as 100%, and two other solutions (200% and 0% iron concentration) were tested. In the commercial product (Iron: 100%), fine zinc particles were deposited uniformly on the aluminum surface. However, the zincate film which was deposited from iron additive-free (Iron: 0%) zincate solution produced a coarse zinc deposit, and aluminum was not covered completely. On the other hand, the zincate film deposited from the double iron concentration (Iron: 200%) had many zinc particles deposited. Moreover, the amount of zinc deposit in the zincate film tended to increase when the iron concentration of the zincate solution increased. The cross-section result by FIB after plating 5μm of electroless nickel film is shown in Fig. 8. Although aluminum spikes could not be observed from iron additive-free and a commercial product, the spike could be observed from double iron concentration. As shown in Fig.9, when the strength of nickel bump shear was measured instead of adhesion, the shear strength of iron additive-free zincate film was the lowest. Moreover, although adhesion strength increased when using the solutions containing iron, even when iron concentration was 200%, there was no difference in the strength of nickel bump shear. The element depth profile analysis after the second zincate deposit from the commercial zincate solution is shown in Fig.10. From analysis of the depth profile, since iron concentration shows the maximum in the region with the interface between the zincate deposit and aluminum, it is believed that immediately after zincate immersion, iron deposits on the aluminum surface preferentially and acts as a zinc deposit site. From the

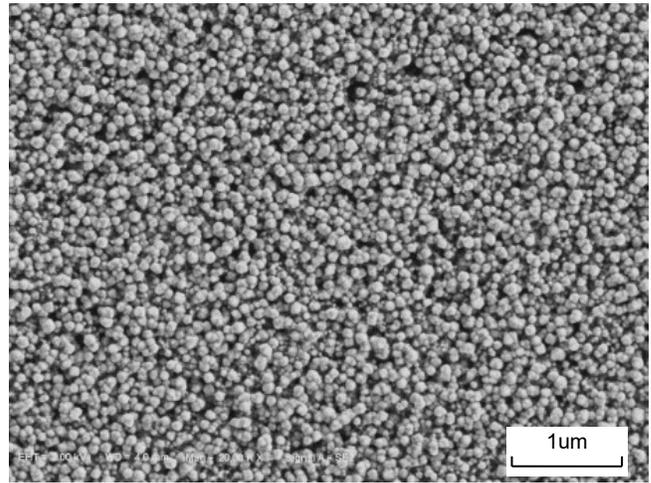
above result, it is considered that the existence of iron in zincate solution has influenced one cause of a spike to aluminum. In other words, aluminum near the deposited iron dissolves preferentially and zinc deposits. Therefore, it is considered that the deposit site of zinc increases in tandem with the iron concentration and the corrosion to aluminum was accelerated because aluminum dissolves in these points. Conversely, when a zincate solution with no iron is used, coarse zinc particles are formed because covering by the zinc is incomplete. In addition, iron has a catalyst action to the hypophosphorous acid ion, which is a reducing agent of electroless nickel plating solutions. It is believed that iron contributes to the adhesion between aluminum and electroless nickel. From the above result, iron in the zincate film is indispensable in assuring adhesion between aluminum and nickel. If iron concentration in the zincate film is not controlled properly, spikes will penetrate the aluminum layer.



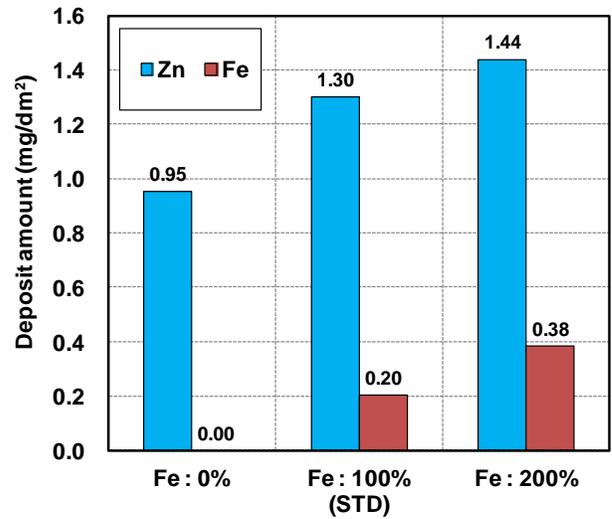
(Fig.6-A) FE-SEM image after second zincate film (Iron additive-free)



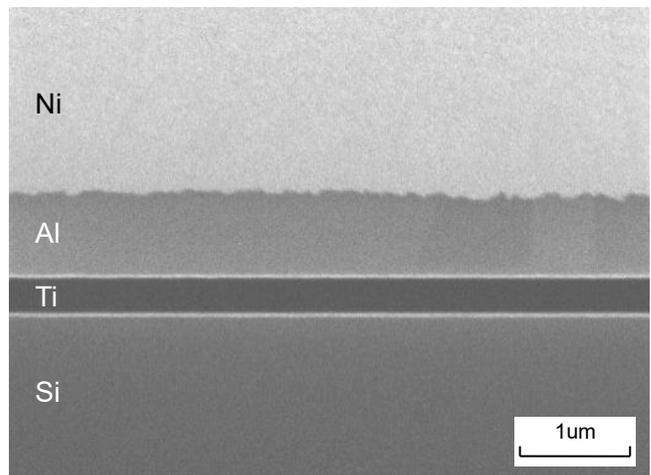
(Fig.6-B) FE-SEM image after second zincate film (Commercial product)



(Fig.6-C) FE-SEM image after second zincate film (Iron: 200%)



(Fig.7) The amount of displacement of zinc and iron in the zincate film



(Fig.8-A) Cross section SIM image after electroless nickel plating (Iron additive-free)

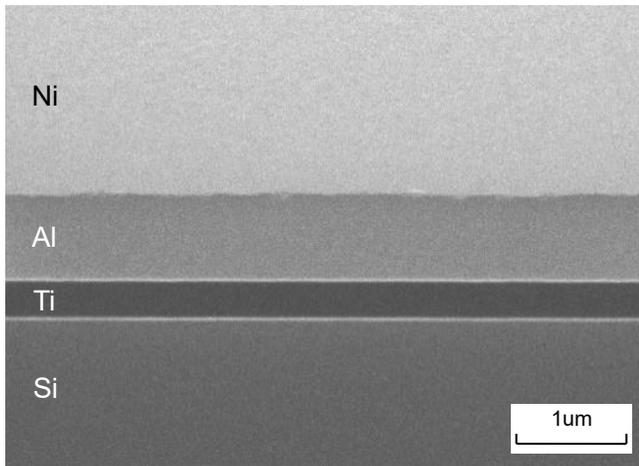
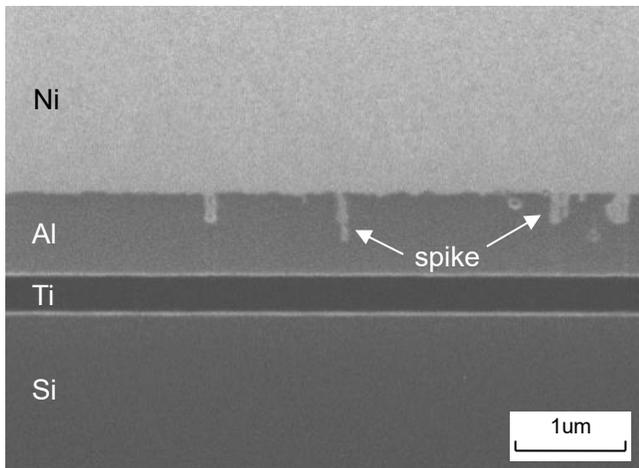
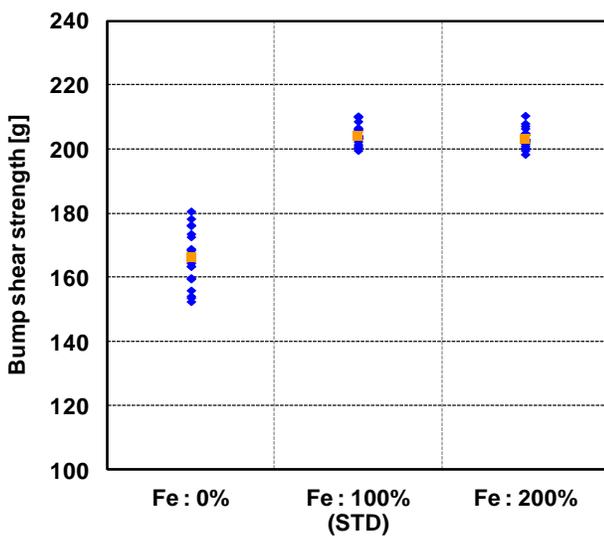


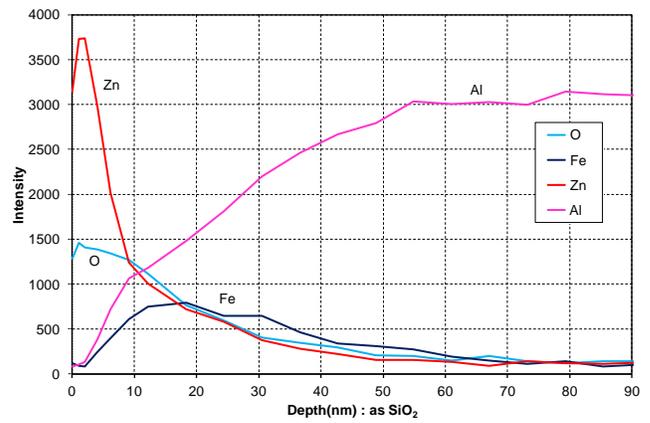
Fig.8-B) Cross section SIM image after electroless nickel plating (Commercial product)



(Fig.8-C) Cross section SIM image after electroless nickel plating (Iron: 200%)



(Fig.9) Strength result of the nickel bump shear test



(Fig.10) Element analysis of the depth profile after the second zincate film that deposited from the commercial zincate solution

### Conclusion

The focus of this study was aluminum pre-treatment prior to electroless nickel plating for the formation of UBM deposits. The etching and zincate process steps were studied to identify possible improvements. We found it was possible to improve the adhesion between aluminum and nickel and to reduce spikes in the aluminum coating. Additionally, completely uniform electroless nickel deposits were possible by optimizing the pre-treatment. Optimizing the etching and zincate process steps led to improved coverage of electroless nickel, excellent adhesion, and minimum spikes into the sputtered aluminum layer.

### Reference

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