

Studies and Applications of EDS, AES and TOF-SIMS Analytical Techniques in Failure Analysis of Fluoride and Chlorine Contamination

Yunan Hua^{1,2*}, Lois Liao² and Xiaomin Li²

¹Wintech-Nano (Nanjing) Co, Ltd Floor 1, Building B, No6, Mingyuan Road, Yuhuatai District, Nanjing, 210012, China

²Wintech Nano-Technology Services Pte Ltd, The Alpha #03-26, 10 Science Park Road, Singapore Science Park II, 117684, Singapore

ABSTRACT

With the rapid development of semiconductor IC design, wafer fabrication and advanced packaging technologies, failure analysis plays a very important role. Past experience tells us that in failure analysis one must ensure the correctness of the analysis results, because wrong data are even worse than no data. As the wrong data, sometimes, may make misleading of the root cause identification. Therefore, in this paper we strongly suggest that we should follow a principle in failure analysis, that is: Yes, means yes; No, don't say no. When analyzing an IC sample there is no detection of a certain contaminating element on the surface of the sample, it does not mean that the contaminating element does not exist! We should carefully make some considerations to review analysis methods used, parameters selected, analysis location, sample penetration and sensitivity etc. To ensure the correctness of the analysis results, in this paper, we will discuss in detail through several real failure analysis application cases, and demonstrate how to use EDS, AES and TOF-SIMS FA techniques correctly.

*Corresponding author

Yunan Hua, Wintech Nano-Technology Services Pte Ltd, The Alpha #03-26, 10 Science Park Road, Singapore Science Park II, 117684, Singapore.

Received: November 24, 2025; **Accepted:** November 27, 2025; **Published:** December 08, 2025

Keywords: Failure Analysis, A FA Principle, EDS, AES, TOF-SIMS

Introduction

With the rapid development of semiconductor IC design, wafer fabrication and advanced packaging technologies, failure analysis plays a very important role. In failure analysis, we know how important it is to provide fast and accurate failure analysis results. In other words, the analysis results must not only meet the needs of customers' R&D and production quickly and timely, but it is more important to provide accurate analysis results.

In failure analysis (FA) of semiconductor IC samples, let us take the analysis of contaminating elements on semiconductor IC samples as an example. As we all know, during the analysis process, we sometimes detect some contaminating elements, then we can write a report saying what contaminating elements we detected; but when during the detection process, we do not detect any contaminating elements, then do we can it be said that there is no contaminating element on the sample analyzed? This is uncertain and incorrect. We haven't detected it in the past, which doesn't mean it doesn't exist. Therefore, in this paper, we will put forward a point of view, that is to say: a principle to be followed in failure analysis, that is: Yes, means yes, No, don't say no.

So why is this? Because when you analyze an IC sample and there is no detection of a certain contaminating element on the surface of the sample, it does not mean that the contaminating element does not exist! Because we know that when analyzing and detecting IC samples, whether contaminating elements can

be detected depends on many factors, such as:

- Choice of FA Techniques
- Choice of FA Parameters
- Choice of FA Locations
- Choice of FA Penetration
- Choice of FA Sensitivity, etc.

In other words, sometimes contaminating elements are not detected, which may be due to because the selected analysis technique is not suitable; it is also possible that the selected analysis method is not sensitive enough; or it may be that the analysis point/position on the IC sample is wrong, etc.

Past experience tells us that in failure analysis we must ensure the correctness of the analysis results, because wrong data are even worse than no data. Therefore, we strongly suggest that we should follow a principle in failure analysis, that is: Yes, means yes; No, don't say no. In this paper, we will discuss and demonstrate in detail through several failure analysis application cases.

Choice of FA Technique

Let's take two analysis techniques of EDS and AES as examples for easily discussion.

Energy Dispersive X-Ray Spectroscopy (EDS or EDX) is a bulk analysis technique (Figure 1). The e-beam/sample interaction area is bigger than the spot size. The penetration depth is related to the beam acceleration voltage used and elements interested. We can estimate the penetration depth using the Monte Carlo electron flight simulation method [1]. The penetration could be

at 0.3-8.5um level dependent on the beam acceleration voltage (kV) and elements analysed. Moreover, EDS is more sensitivity to high atom Z elements (metallic elements) and the detection limit is about 0.1-1.0 at% [2-5].

However, Auger electron spectroscopy (AES) is a surface analysis technique. The e-beam/sample interaction area is same as the spot size and the penetration depth of AES is only 10-20 Å (Figure 1). AES is more sensitivity to low atom Z elements (such as C, O, F etc.). Its detection limit is similar to EDS's about 0.1-1.0 at%.

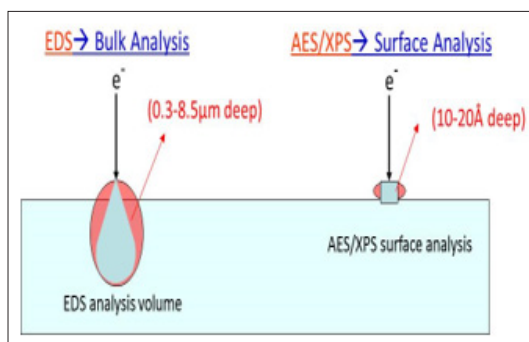


Figure1: EDS is a Bulk /Volume Analysis Technique and the Penetration Depth is much Deeper to 0.3-8.5um. But AES is a Surface Analysis Technique and the Penetration Depth is only 10-20 Å

Although the detection limit of both EDS and AES is similar about 0.1-1.0 at%. However, EDS and AES techniques are much different from each other as EDS is a bulk analysis on sample and the penetration depth is about 0.3-8.5um, but AES a surface analysis on sample and the penetration depth is only about 10-20 Å. Therefore, even if for the same sample, the EDS and AES analysis results will be much difference.

For example, in wafer fab, there are 2 samples (S1 and S2) with relatively high F (S1) and low F (S2) contamination on Al bondpads. In order to determine F contamination level on surface of Al bondpads for both samples, in the first, we used EDS to do analysis and the results showed that no F was detected on the both samples. Then, based on EDS results can we say no F contamination on Al bondpads for both samples? The answer is no because EDS is a bulk analysis on sample and the penetration depth is about 0.3-8.5um. But F contamination is usually on very surface (about 1-2nm) of Al bondpads, and now during EDS analysis it is diluted to a depth of microns level, which is diluted about 10,000 times. The amount of F contamination detected is much lower than the detection limit of EDS technique and EDS cannot detect F contamination. So, the conclusion is that we should not choose EDS, but should choose AES to analyze F contamination on the surface of Al bondpads.

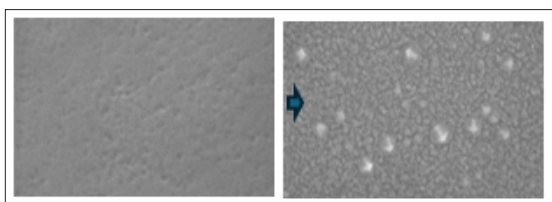


Figure 2: S1 is Bad Sample: AES Result showed that High F (about 8.5 at%) on the Al Bondpad, which is Higher that the control limit of 5.0 at %. There were lot of the “Crystal-like” Defects on Al Bondpad after SLAT Test ((Left: Before SLAT Test and Right: After SLAT Test)

Then, we employed AES to do analysis. AES results showed that the F contamination level is 8.5 at% on Al bondpad of the S1 sample and it is 3.3 at% on Al bondpad of the S2 sample, respectively. In our previous studies, we have studied and setup the control limit of the F contamination on a normal Al bondpad, which is 5.0 at% using AES analysis. Therefore, based on the AES analysis results, one can justify that the S2 is good Al bondpad as its F contamination level is only 3.3 at%, which is lower than the control limit 5.0 at%. Based on AES results, we could see that sample S1 was out of control and failed as its F contamination level on Al bondpad is 8.5 at% which was higher than the control limit 5.0 at%. So S1 is identified as a bad sample. Our SLAT test result (Figure.2) also showed that there were lot of the “crystal-like” defects on Al bondpad after SLAT test, which might cause NSOP problem during assembly process.

Based on AES results, we could see sample S2 was within control and passed as its F contamination level on Al bondpad was 3.3 at% which is lower than the control limit 5.0 at%. So S2 is a good sample. Our SLAT test result (Figure.3) also showed that there was no defect on Al bondpad after SLAT test.

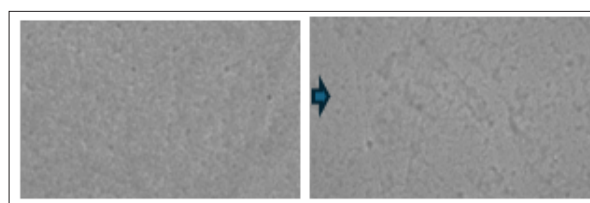


Figure 3: S2 is Good Sample: AES Result Showed that Low F (about 3.3 at%) on the Al Bondpad, which is Lower that the Control Limit of 5.0 at %. There was no Defect on Al Bondpad after SLAT Test (Left: Before SLAT Test and Right: After SLAT Test)

From this case, one can see that AES analysis could help us to identify F contamination level on S1 and S2. But EDS could not achieve it. Why? From Figure.1, we can know that the EDS's penetration is much deeper to 0.3-8.5um. If we assume that EDS's penetration depth is about 1um and AES's penetration depth is about 20Å (2nm), and then for the S1 and S2, we can easily estimate and calculate the EDS results according to the AES results:

$$\text{S1: EDS (S1)} = (8.5\text{at}\% \times 20\text{\AA}) / 10000\text{\AA} = 0.017 \text{ at}\% \quad (4)$$

$$\text{S2: EDS (S2)} = (3.3\text{at}\% \times 20\text{\AA}) / 10000\text{\AA} = 0.0066 \text{ at}\% \quad (5)$$

Moreover, we have known the detection limit of EDS is about 0.1-1.0 at%. The above both values from Eqn (4) and Eqn (5) are much lower than the EDS's detection limit (0.1 - 1.0 at%). Thus we can fully understand why EDS cannot detect and identify the F contamination levels of the S1 from S2.

From this case discussed above, we can fully understand that EDS does not detect F contamination, which does not mean that there is no F contamination on the Al bondpads. We should apply surface analysis technique such as AES to measure F contamination on Al bondpads. Moreover, we also understand that we can't directly compare EDS results with AES results in the future. A practice rule is proposed and must be followed-up that “EDS clean is not clean” and “Auger clean is clean”. At the moment, we fully recommend not to use EDS, but use AES to analyze and monitor surface F contamination level on a normal Al bondpad in wafer fabrication.

Choice of FA Parameter

In this section, let's still take EDS analysis as examples for further discussion. As we all know, when doing EDS analysis, it is very important to choose the appropriate beam accelerating voltage. Because the beam accelerating voltage is selected as lower, we may cause insufficient excitation energy and some elements of interest are not excited, thus missing some contaminating elements. On the contrary, if the beam accelerating voltage is selected higher, the penetration depth will deepen, and elemental information from the underneath layers of the sample will be detected, causing confusion in the analysis results.

In this FA case, it was reported that the unit failed functional test during multi-port testing. After decapsulation, EMMI was performed on the failure unit and found the hot spot. The unit was performed delayering and X-sectional process. FIB/SEM results showed that a particle was the above metal 1 layer (Figure 4, SEM). EDS analysis was done on the particle using 5kV beam acceleration voltage, the EDS result showed that C, O, F, Ga, Al, Si and Pt peaks (Figure 4, EDS), in which Ga was introduced by FIB sample preparation and Pt was from sample Pt coating.

Based on the EDS (5kV) results (Figure. 6, EDS), one can see that no iron (Fe) was detected. In this situation, can we conclude no Fe contamination in the particle? The answer is no. This is because the EDS energy of 5kV beam acceleration voltage cannot excite Fe ka peak (6.403 keV) [3].

Although Fe La peak (0.705 keV) could be excited, but it is overlapped with F Ka peak (0.677 keV). As such we cannot identify Fe La peak from F Ka peak due to energy resolution. Therefore, to make further confirmation, we have to do EDS analysis using higher beam acceleration voltage.

We did EDS analysis again on the particle using 15 kV and the results are shown in Figure 5. From it one can see that Fe Ka peak at 6.403 keV is detected. Moreover, besides Fe Ka peak, Cr Ka and Ni Ka peak were also detected. Thus, based on EDS (15kV) results, we could confirm that the peak detected at 0.677 keV was not F Ka peak, but was Fe La peak. Therefore, the particle was not a fluorine contamination related, but most likely to be a stainless steel particle as Cr, Ni and Fe were detected together.

From this case, we fully understand that it is very important to select correct experimental parameters, such as suitable beam acceleration voltage [3].

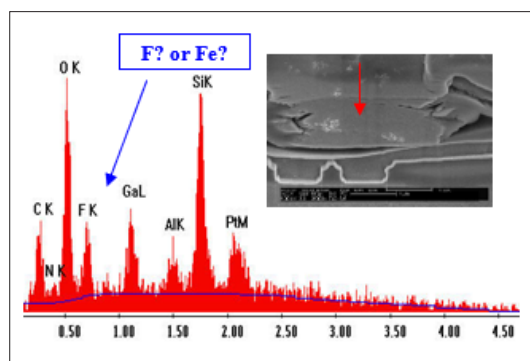


Figure 4: SEM/ EDS Analysis was done on the Particle Using 5kV Beam Acceleration Voltage and EDS Result Showed that C, O, F, Ga, Al, Si and Pt Peaks, in which Ga was Introduced by FIB Sample Preparation and Pt was from Sample Pt Coating

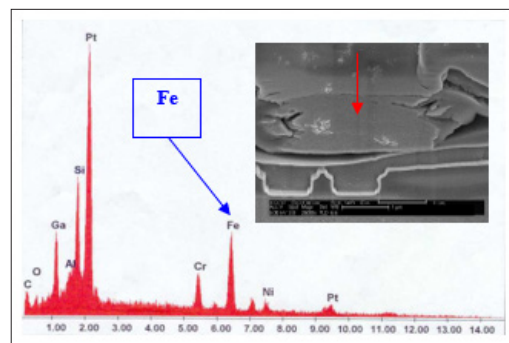


Figure 5: We did EDS again on the Particle Using 15 kV and EDS Results Showed that Fe Ka Peak at 6.403 keV was Detected

Choice of FA Location

It is well known that in FA, most time, the contamination is not uniform, but localized on the sample. Sometime, we may miss to detect some contaminants. For example, Figure 6 shows a sample with white color contamination (left side). To detect the contaminating elements, we did point-EDS analysis on the white color contamination area. The EDS result showed that fluorine (F) contamination was detected.

Based on the point-EDS analysis results, we can say certainly that F contamination was detected on the sample. But EDS did not detect Chlorine (Cl) element, so can we say that there is no Cl contamination? The answer is no as not detected does not mean nothing.

Then, we applied TOF-SIMS color mapping analysis technology to analyze the sample and detected both F and Cl contamination on the sample. It's just that the Cl contamination area was located at the edge of the sample, making it difficult to detect and miss during point-EDS analysis.

From the analysis results of TOF-SIMS color mapping, we can catch all the contaminating elements without missing. This fully demonstrates the superiority of TOF-SIMS color mapping analysis technology. It also fully proves the principle to be followed in failure analysis, that is: Yes, means yes, no, don't say no.

For example, in this case, we did not detect the Cl element using point-EDS analysis. This is because the Cl contamination area is located at the edge of the sample, when doing point-EDS analysis, the Cl contaminated area was not chosen, and then the Cl contamination was missed. As such, we think that the choice of FA location is also very important.

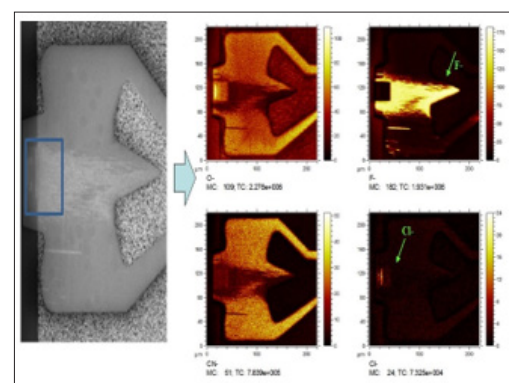


Figure 6: TOF-SIMS Ion Mapping Result shows that the Cl Contamination Area was Located at the Edge of the Sample and the Cl was not Detected using Point-EDS Analysis

Choice of FA Penetration

We know that chlorine (Cl) contamination may greatly impact quality of IC. Figure 2 shows a Cl corrosive defect on the Al metal line. One can see that the defect has damaged the metal line, which could cause low yield or reliability issue. In this case, the root cause of the Cl contamination was identified to be Cl contamination from environment of the wafer fabrication.

Chlorine chemically reacted with Al and formed $AlCl_3$ which is a chemically unstable compound and could dissolve in water. Therefore, Cl-induced corrosion is more serious as it can cause chemical chain reactions [5].

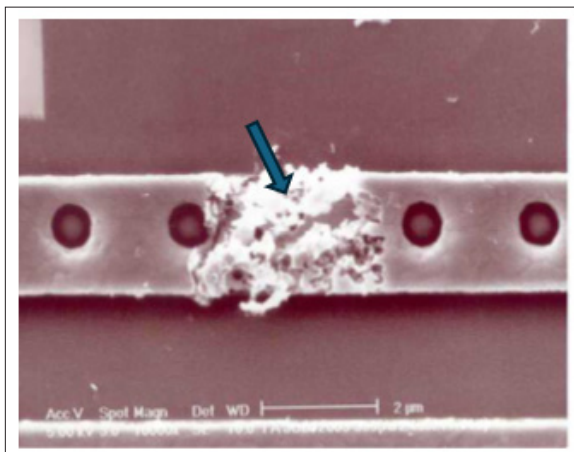


Figure 7: SEM Micrograph Showed the Corrosive Defect on Al Metal Line due to Cl-Induced Corrosion

To understand the contaminating elements, we did FA. At the first, we did Auger Electron Spectroscopy (AES) analysis on the defect area of the sample (Figure.7 shown as the arrow). However, the AES analysis results showed that in addition to measuring carbon (C), oxygen (O) and Al, there was no Cl element was detected as the energy spectrum peak of Cl was not appeared at the AES energy spectrum peak position at 184eV (Figure 8 shown as the arrow).

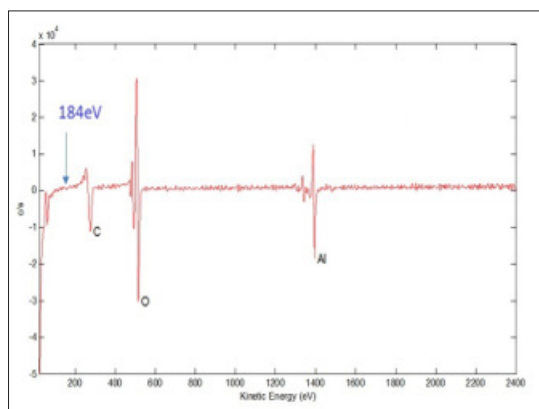


Figure 8: AES Result Showed no Cl Element was Detected as no Peak at AES Spectrum at 184eV

AES not detected Cl on the defect. Does it mean that there is really no Cl contamination in the defect? What contaminating element is the defect in Figure 2 caused by? We should make careful consideration to follow the FA principal: Yes, it is yes! No, cannot say no!

For example, in this case we detected C, O & Al. It is true and we can say: yes, there are C, O and Al in the defect. However, we not

detected Cl and cannot say there is no Cl in the defect.

As such, we also conducted X-ray energy spectroscopy (EDS) on the defects. The EDS (15kV) results showed that in addition to C, O and Al, Cl was also detected (Figure 9). This confirmed that the defect was caused by Cl-induced corrosion. Then we could say yes, as Cl was detected.

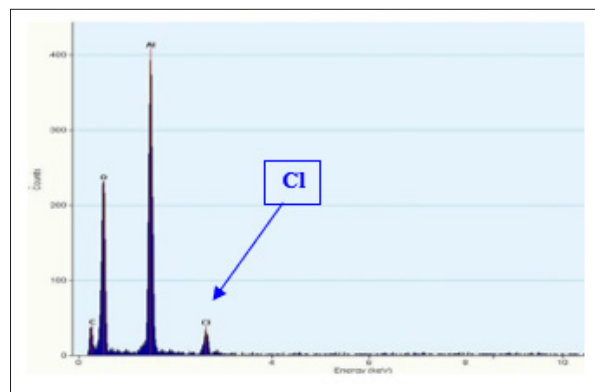


Figure 9: EDS (15kV) Detected Cl Besides Carbon, Oxygen and Al on the Cl-Induced Defect shown in Figure 7

Based on analysis results from both ARE and EDS, we found much difference as: AES no detected Cl, but EDS detected Cl. Why AES and EDS were with different results? This is because AES is a surface analysis and EDS is a bulk analysis. Their analysis penetration depths are much difference. From Figure. 1, we can understand that EDS is a bulk analysis and the penetration depth is about 0.3-8.5um, but AES a surface analysis and the penetration depth is only about 10-20 Å.

Another side, Cl compound is a chemically unstable that is easily soluble in water. Therefore, in this case, the Cl on the defect surface has been washed away by the cleaning steps in wafer fab. This resulted in the fact that we did not detect Cl on the defect surface when we used AES. This is because AES is a surface analysis technique. So, no Cl was detected on surface of the defect.

However, EDS is a bulk analysis technique; the penetration depth is much deeper and can detect Cl inside of the defect.

As such, in the case, if we conclude based on the AES analysis results that there is no Cl contamination, it would be a big mistake.

Choice of FA Sensitivity

In the above FA case, using AES, no Cl was detected as Cl on the defect surface might be washed away by the cleaning steps in wafer fab. Then we could use EDS to analyse Cl contamination inside of the defect and to confirm the present of the Cl contamination.

In this section, we would continue to discuss choice of FA sensitivity. We know that AES's detecting limit is about 0.1-1.0 at %, but TOF-SIMS's detecting limit is PPM. Therefore, for the defect in Figure x, we could not detect Cl on surface of the defect. This is because the amount of the Cl contamination might be lower than EDS's detection limit. However, if we use more sensitivity analysis method, such as TOF-SIMS, we might detect low level Cl contamination.

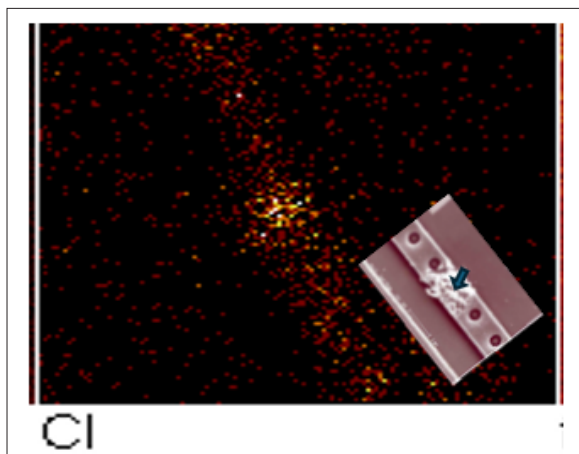


Figure 10: The Result from TOF-SIMS Ion Mapping Analysis showed that Cl was Detected on the Defect

Conclusion

In this paper, we put forward a point of view, that is to say: a principle to be followed in failure analysis, that is: Yes, means yes; No, don't say no. When analyzing an IC sample there is no detection of a certain contaminating element on the surface of the sample, it does not mean that the contaminating element does not exist! We should carefully make some considerations to review analysis methods used, parameters selected, analysis location, sample penetration and sensitivity etc. To ensure the correctness of the analysis results, in this paper, we have discussed and demonstrated in detail through five real failure analysis application cases.

Acknowledgment

The authors would like to thank personals from Win tech-Nano for providing technical support and making contributions to this technical paper.

References

1. Hua Younan, Guo ZR, Chau KW (1997) Studies of ZAF Standardless EDX Quantification Method and Application in Failure Analysis of Semiconductor. Journal of Trace and Microprobe Techniques 15: 13-31.
2. Hua Younan (1999) Studies of EDX ZAF Quantification Method and Characterization of EDX Function in LEO 982 FESEM Equipment. The Proceedings from the SEMICON, Singapore, Testing, Assembly and Packaging 99-Technical Program 207-212.
3. Hua Younan (2004) Studies on An Estimating Method for Electron Beam Acceleration Voltage Used in Energy-Dispersive X-Ray Microanalysis Technique and Its Application in Failure Analysis of Wafer Fabrication. Journal of Instrumentation Science and Technology 32: 115-126.
4. Hua Younan, LIU Binghai, MO Zhiqiang, TEONG Jennifer (2008) Studies and Applications of Standardless EDX Quantification Method in Failure Analysis of Wafer Fabrication. The Proceedings of the 15th International Symposium for on the Physical and Failure Analysis of Integrated Circuits, Singapore 15: 290-295.
5. Hua Younan, Liao Jinzhi, Liu Binghai, Zhu Lei, Li Xiaomin (2023) Failure Analysis & Mechanism Studies of the Worm-like Defects in Vias of Wafer Fabrication. The Journal of Engineering Research and Sciences 2: 1-6.

Copyright: ©2025 Younan Hua, et al. This is an open-access article distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited.