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TECHNICAL ARTICLE—PEER-REVIEWED

Failure Analysis of Un-Wetting for the Surface Finish on the ENIG

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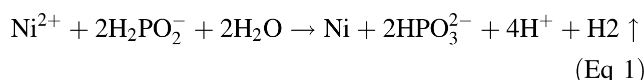
Abstract Electroless nickel immersion gold (ENIG) is one of the most prevailing surface finishes for printed circuit board. It is widely adopted by manufacturers all over the world for its relative low price and compliance with the trend of high density packaging. However, its reliability is always of a concern. In this paper, the poor wetting performance of tin solder on the ENIG surface, which is one of its reliability issues was addressed. A series of modern facilities were utilized for finding the failure mechanism and chasing down the root cause. It was concluded that the inferior quality of immersion gold layer during plating, to be more specifically, the coarse and big grain size and the crystalline interface cracking were the main causes for the un-wetting failure. Last but not least, the improper choice of pure tin solder should also contribute to the final failure.

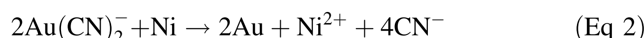
Keywords ENIG · Un-wetting · Grain size · Failure analysis · Oxidation

Introduction

Printed circuit board (PCB) is the foundation of all kinds of electronic devices ranging from cell phone, personal computer to military products. With the booming development of integrated circuits (IC), there is increasingly stringent requirements placed on reliability of PCB [1, 2] since it is the platform which connects all the chips together and provide the interconnection between them. The surface finish of PCB is one of the key factors which has a significant influence on its reliability performance. As illustrated in Fig. 1, surface is the bonding bridge of die and substrate. Nowadays, electroless nickel immersion gold (ENIG) is the most popular surface finish adopted by nearly all the PCB manufacturers around the world [3]. The primary process of ENIG is to deposit a layer of nickel and then a layer of gold onto the PCB substrate, as exhibited in formulae (1) and (2). According to IPC-4552 standard, the nickel layer should be between 3- and 6-μm thick and golden layer be 50–100-nm thick to ensure its reliability. The specific thickness of ENIG should be determined by special circumstance in which the product will be applied. The golden layer is employed for maintaining the solder ability during manufacturing process like reflow and wave soldering. In some situation, such as Golden Finger, the golden layer is utilized for providing anti-corrosion and abrasion resistance property. In another application, manufacturer will bond aluminum or golden wires onto the golden surface to realize the electric interconnection between the PCB and outer circuit. Nickel is used as a barrier preventing atoms in the substrate material from diffusing into the golden layer and forming intermetallic compound (IMC).



$$Ni^{2+} + 2H_2PO_2^- + 2H_2O \rightarrow Ni + 2HPO_3^{2-} + 4H^+ + H2\uparrow \quad \text{(Eq 1)}$$



$$2Au(CN)_2^- + Ni \rightarrow 2Au + Ni^{2+} + 4CN^- \quad \text{(Eq 2)}$$

In this paper, a batch of PCB sample finished with ENIG was found to display poor wetting performance after hot air solder leveling. For the purpose of finding the failure mechanism, locating the root cause and finally improving reliability, a series of modern analytical instruments such

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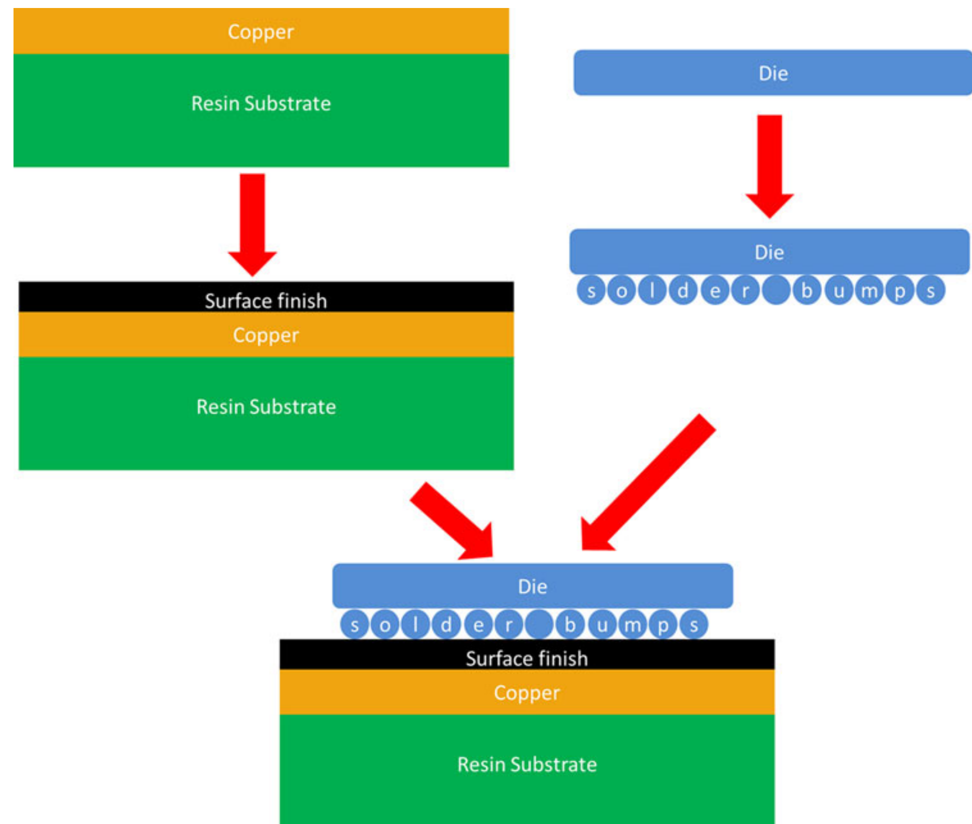


Fig. 1 The role of surface finish in PCB

as 3D stereo microscopy, scanning electron microscope (SEM), and energy dispersive spectroscopy (EDS) were adopted. The un-wetting mechanism and the root cause of the failure were illustrated. Counter strategies and suggestions were given in the end.

Sample and Experiment

Figure 2a is the plane configuration of the failed PCB which has a dimension of 1.2 cm × 2.4 cm. The sample was treated with ENIG surface finish, then desmeared, and subsequently went through hot air solder leveling. The little squares on sample are pads where tin solder should be attached on. Under optical inspection, as Fig. 2a shows, some pads are emitting shiny silver light and these are the ones soldered appropriately. While some of the pads just appear golden, and these are the ones not properly wetted by tin solder, thus letting the underlying golden layer exposed to the environment, displaying a color of golden to our naked eye. Figure 2b is the corresponding schematic diagram of the failed sample which clearly shows that there are 8 out of 129 pad un-wetted which is not endurable.

In order to dig out the primary cause of the failure, modern analytic instruments and characterization methods have been adopted. The de-wetting pad was observed by SEM, and its chemical composition was analyzed by EDS. One well-wetted pad was also analyzed by EDS for contrast and comparison.

Results and Discussion

A section of failed pad indicated by the red square in Fig. 2a was chosen for SEM inspection. The reason for choosing this section is that it contains both the well-wetted and de-wetted pads, thus the contrast is distinct.

As Fig. 3a shows, although the normal and failed pads all appear gray under SEM, there is still observable difference between the two parts. The normal (well-wetted) pad displays a bulgy surface topography, while the failed (de-wetted) pad displays completely flatness. This is because tin solder will form a bump on pad under the effect of gravity and capillary force, rendering the normal pad a bulgy profile. In a similar way, since the de-wetted pad has no solder attached on it, its outermost surface is actually the final finish of ENIG which accounts for its flatness.

The failed pad which is indicated by the red circle in Fig. 3a was further magnified. As it can be seen in Fig. 3b, the grains of the immersion gold on the failed pad are clear and it appears coarse under magnification of 2,000×. This feature became more obvious when the spot was magnified to 4,000× (Fig. 3c). When magnified to 16,000×, besides

Fig. 2 Macroscopic appearance of the failed sample. (**a**) Plane configuration of sample, (**b**) schematic diagram of sample

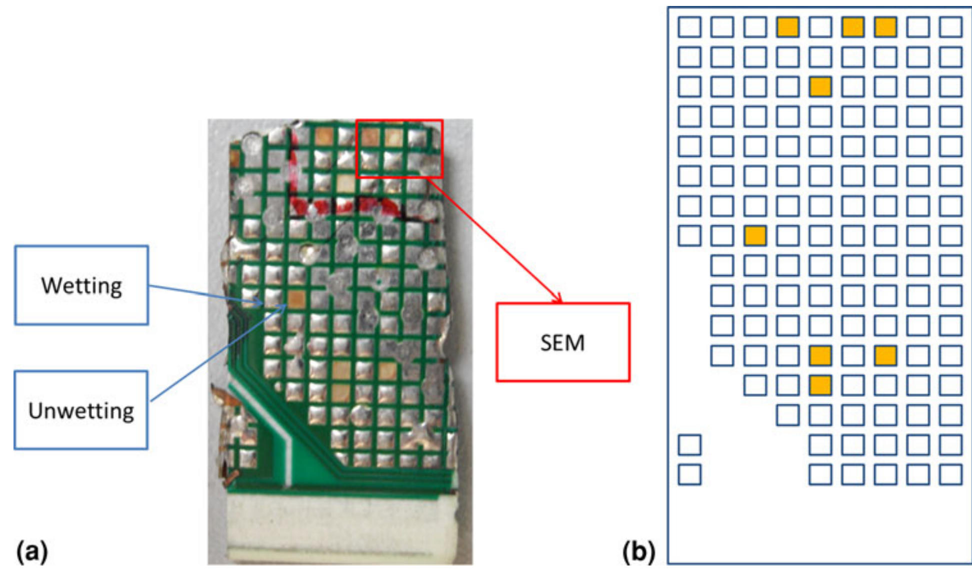


Fig. 3 SEM results of the un-wetted pad. (**a**) Un-wetting pad displays flatness, (**b**) coarse grains under 2,000×, (**c**) coarse grain under 4,000×, and (**d**) crystalline interface cracking

the coarse topography, we can also indentify crystalline interface cracking as labeled in Fig. 3d. It can also be estimated that the cracking length is about 3–4 μm.

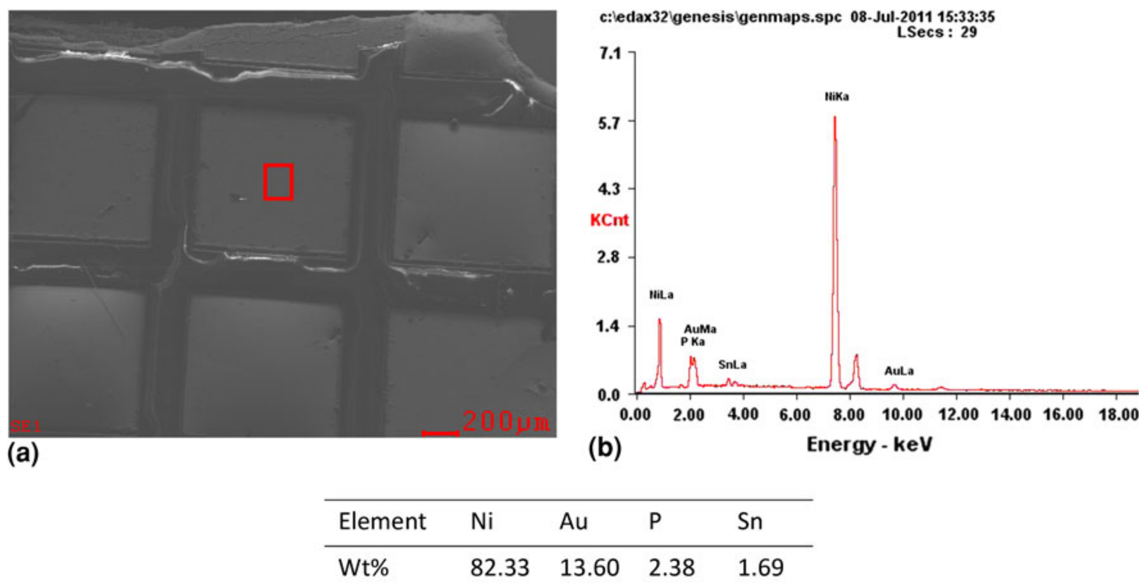
The region marked by a red square on failed pad was chosen to be analyzed by EDS. The chemical composition and corresponding weight percentage is given in Fig. 4. As it illustrates, four detected elements are Ni, Au, P, and Sn. And the weight percentage is 82.33, 13.60, 2.3, and 1.69%, respectively. It is reasonable to find Ni and Au, since the surface finish is ENIG. The existence of P is acceptable because phosphorus was introduced in during chemical plating of nickel. The little quantity of Sn on failed pad could be explained as follows: although the pad was not well-wetted, there was still some tin solder residue on it after been through hot air solder leveling.

In order for comparison, EDS analysis was also utilized on a section of a normal (well-wetted) pad of the same sample as show in Fig. 5. Likewise, the information of chemical composition and weight percentage were revealed. From Fig. 5, we mainly found Sn with little quantity of C and O. This result confirmed us that solder used for joint was pure tin and C and O were organic contamination from the surrounding environment which accidentally adhered to tin bump during storage.

According to the formula, one nickel atom will be replaced by two gold atoms. The radius of nickel and gold atom is 1.24 and 1.44 Å, respectively, thus there is an atomic radiuses difference of 16% between Ni and Au. The synergy of the two factors above left the ENIG finish a rough bumpy surface full of pits and holes as illustrated in Fig. 6. These pits and holes then exposed the nickel straight to oxygen in the air and caused it to form nickel oxide (Fig. 7) whose stoichiometry is $Ni_{29}O_{71}$ according to Chong [4], or as Lee [5] proposed in his work, the nickel oxide displayed stoichiometry of NiO_2 in the outermost part and NiO in the inner part. No matter how different the stoichiometry of nickel oxide is, it ultimately acted as an obstacle and blocked off the tin solder from contacting the gold layer under it during hot air solder leveling. Since nickel oxide is a relatively stable substance, the desmear before soldering could hardly play any role. This explains why de-wetting still could not be avoided even after careful desmear had been implemented before soldering.

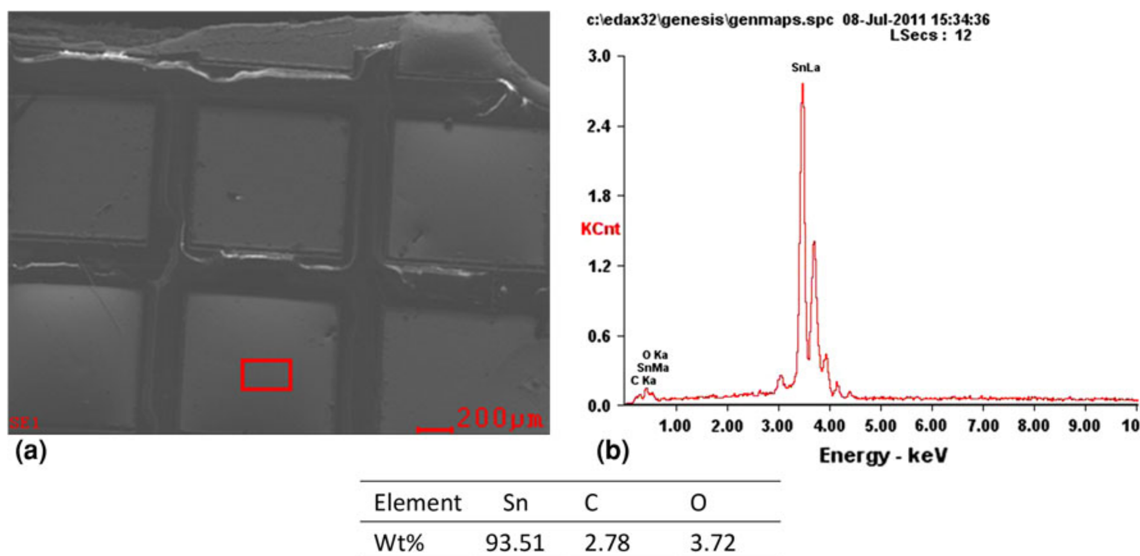
As revealed by the SEM result (Fig. 3d), the existence of crystalline interface cracking acted as a "Grand Canyon", thus aggravated this mechanism by exposing more underlying nickel atoms to oxygen in the environment. Moreover, through "Grand Canyon", the nickel atoms lied below diffused upward through the canyon to the outermost surface, covering the gold layer and forming a sandwich structure finally. Then the external nickel layer was oxidized and formed a barrier between tin solder and gold layer during hot air solder leveling later. The process described above was demonstrated in Fig. 8.

Besides that, organic substance which is highly volatile from the depositary environment was absorbed into the canyon and covered the nethermost nickel layer, acting as an isolating layer between solder and nickel and led the un-wetting during soldering. Since canyon had a considerable absorption capacity and as proved by Fig. 5 that the quantity of organic contamination in storage circumstance could not be neglected, this mechanism was equally possible for the poor wetting performance. Figure 9 schemed out the process. However, it is necessary to be put out that



Element	Ni	Au	P	Sn
Wt%	82.33	13.60	2.38	1.69

Fig. 4 EDS result of the un-wetting pad. (**a**) The un-wetting pad, (**b**) chemical composition, and (**c**) weight percentage of main elements



Element	Sn	C	O
Wt%	93.51	2.78	3.72

Fig. 5 EDS result of the wetting pad. The chemical reaction formula of depositing Au on nickel layer is as follows: $2Au(CN)_2^- + Ni \rightarrow 2Au + Ni^{2+} + 4CN^-$. (**a**) The wetting pad, (**b**) chemical composition, and (**c**) weight percentage of main elements

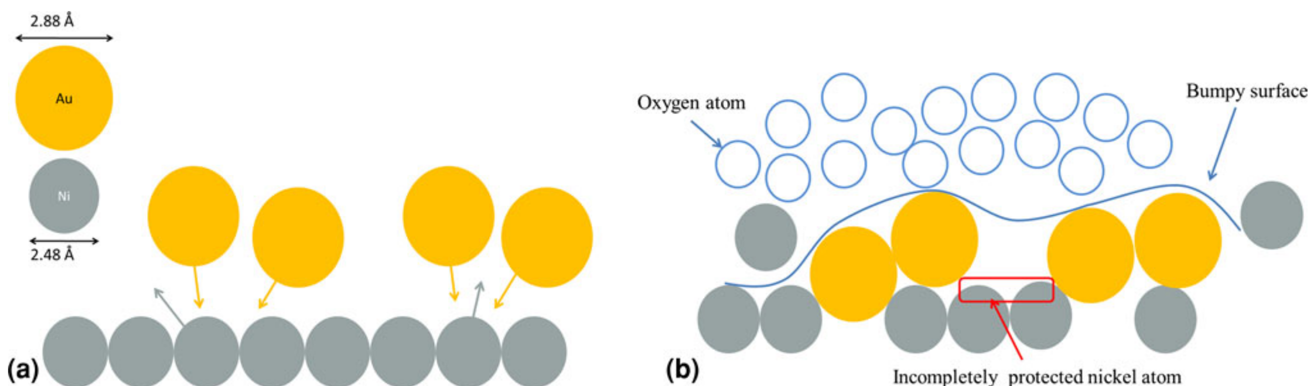


Fig. 6 Formation of bumpy surface during immersion gold. (**a**) The deposition of Au on Ni, (**b**) bumpy surface formed

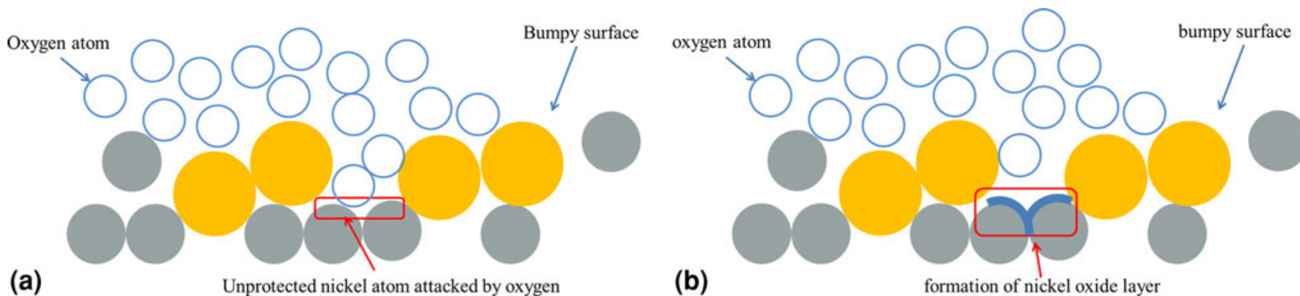


Fig. 7 Formation of nickel oxide layer. (**a**) Oxygen contacts the unprotected Ni. (**b**) The unprotected nickel were oxidized

within our reach there is no device could directly detect or observe such little amount of organic contamination.

As mentioned before (Fig. 3), the grain size of the failed pad under inspection of SEM was relatively coarse and big (3–4 μm). The big grain size led to the smaller total grain boundary area as illustrated in Fig. 10. It is the fact that energy along the grain boundary is higher than that in bulk [6]. Since the atoms alone the grain boundaries were not perfectly bonded, they must have a higher tendency to engage in a chemical reaction compared with

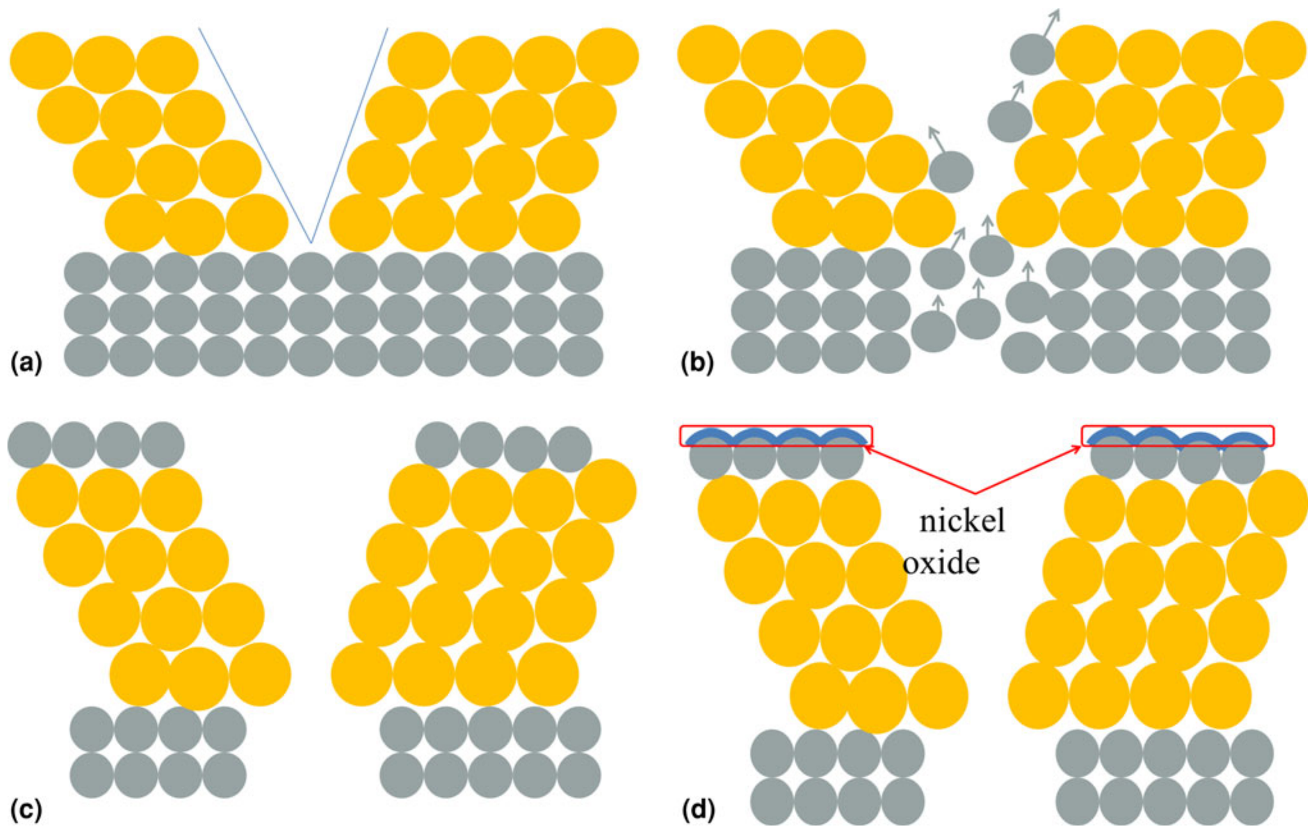


Fig. 8 Grand Canyon mechanism. (**a**) Crystalline interface cracking, (**b**) the diffusion of nickel atom, (**c**) nickel atom covered the outmost surface, and (**d**) nickel oxide formed covering the surface

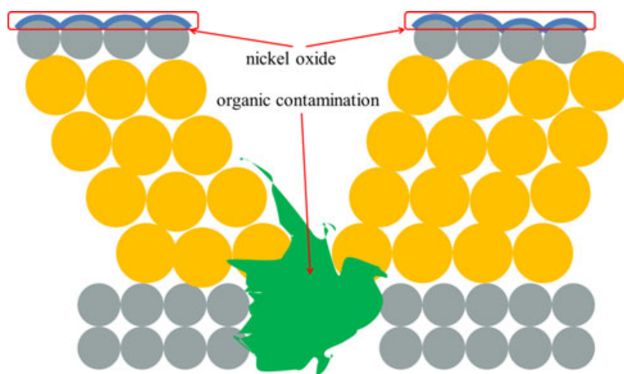


Fig. 9 Organic contamination

those inside the grain. Thus, the reduction of total grain boundary area simultaneously decreased the whole energy of the golden layer. Therefore, the surface golden layer was left in a condition of relatively low free energy. When the sample was sent for soldering, the energy provide by hot air solder leveling was not high enough to fuse the golden layer, since the latter was in an unexpected low energy condition. This inability of smelting the golden layer caused by energy inadequacy manifested itself as un-wetting failure mode. As for the definition of fine grain size, till now there is no direct mathematic function describing the relation between the surface energy and grain size within our knowledge, and it is merely impossible to observe the well-wetted golden layer since it was consumed once wetted. This is a field which needs more research work to be done. Here we proposed that under permitted condition, the finer the grain the better the wetting performance.

Last but not least, the solder used which is pure tin also contributed to the final un-wetting failure. Since pure tin is a relatively obsolete solder and has lower surface energy and higher melting point compared to prevailing solder like Sn–Ag–Cu [7], it cannot be ruled out that it was the improper choice of pure tin solder that resulted in the ultimate un-wetting. A discussion of the promising solder in manufacture of PCB would be beyond the scope of this paper. Abtew and Selvaduray [8] gave a comprehensive introduction of this topic in their excellent paper.

Conclusion

- SEM inspection and EDS analysis were both conducted on the normal (well-wetted) and failed (un-wetted) pad of the same sample. The topography of the two kind of

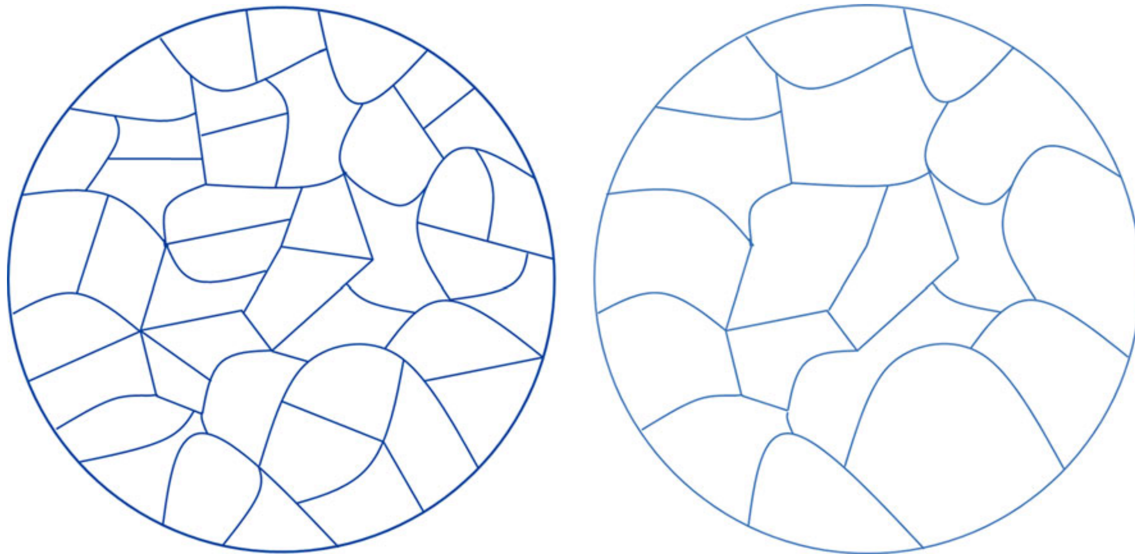


Fig. 10 The relation between grain size and grain boundary area. (**a**) Fine grain thus more grain boundary area, and (**b**) coarse grain thus less grain boundary area

pad was different. Normal pad displayed a bump surface and failed pad displayed total flatness. The failed pad also revealed coarse and big grain size and even crystalline interface cracking under high magnification. The EDS analysis on normal pad assured us that the solder used was pure tin and there was observable quantity of organic contamination during depository.

- In the chemical reaction of ENIG, one nickel atom was replaced by two gold atoms. This fact combined with another fact that there is 16% difference in atomic radius between nickel and gold led to the misalignment of golden layer on nickel layer, forming a rugged surface full of pits and holes. Furthermore, the crystalline interface cracking confirmed by SEM formed "Grand Canyon" from a microscopic view. All these facts described above exposed nickel to air and brought about the formation of nickel oxide which further acting as a barrier between solder and golden layer later during hot air solder leveling. Moreover, the "Grand Canyon" provided a channel for the upward diffusion of nickel atom to the outermost surface and then be oxidized as a solder barrier. The "Grand Canyon" also absorbed organic contamination from surrounding environment into it playing a role as an obstacle for solder reaching nickel.
- The coarse and big grain size revealed by SEM aggravated the un-wetting. The big grain size corresponds to the reduction of total grain boundary area which possessed higher energy than that in bulk, thus lowering the energy of golden layer as a whole. So the predetermined soldering energy from hot air solder leveling was not enough to fuse the golden layer. The inadequacy of energy to melt the surface layer exhibited itself as un-wetting failure mode.
- The pure tin solder used as confirm by EDS analysis on normal pad was inappropriate. Since pure tin has lower surface energy and higher melting point compare to prevailing solders.

Suggestion

- It is proposed that the quality of the plating gold should be improved by adjusting the plating temperature, pH value, solution composition in the plating bath and the stirring mode. Thus, refining the grain size and suppressing the crystalline interface cracking.
- Replacing the pure tin with new solders like Sn–Ag–Cu is strongly recommended.

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