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Copper Crystal Structures in Plated Microvias. Their Recrystallisation and a Means to Identify Joints at Risk of Premature Failure

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Abstract

Plated microvias are widely used within todays PCB industry as a means of achieving the high-density designs that are required in modern mobile devices, however, there has been growing concern regarding their long term reliability performance when stacked directly on top of each other.

Blind microvias (BMV) have a potentially complex metallurgical structure, with several interfaces located around the target pad - electroless Copper - electrolytic Copper joint. While field experience as shown that there are typically two major types of crystal structures formed across the BMV base, there has been little reported work investigating how or why such structures develop. In this paper, we review these two commonly observed microstructures within filled BMVs and offer proposals on how such structures are created. We subsequently describe a novel means to indicate if the microstructure of a BMV is likely to have a tendency for an early onset of failure.

Key words

Recrystallisation, BMV, Reliability, Microvia.

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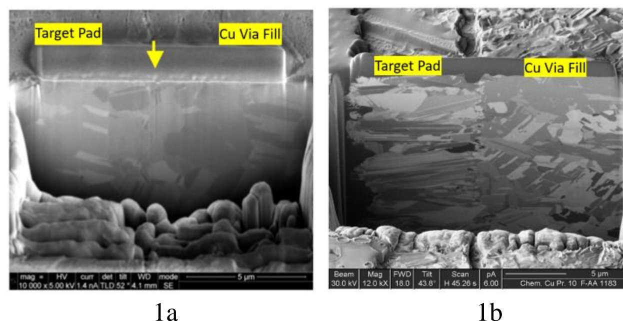
I. Introduction

Blind microvias (BMV) have potentially complex metallurgical structures, with several interfaces located around the target pad - electroless Copper - electrolytic Copper joint. While field experience as shown that there are typically two major types of crystal structures formed across the BMV, and there have been multiple works describing testing, and the subsequent failure analysis [1,2,3] there has been little reported work investigating how or why such structures develop.

Figure 1 shows the typical structures noted in BMVs during routine failure analysis, with figure 1a exhibiting a fully epitaxial structure with no clear delineation between the target pad and the plated Cu, while figure 1b shows a clearly visible interface. As the epitaxial structure is considered "ideal" with no obvious defects, it is taken that this would offer a higher level of structural integrity compared to that shown in Fig 1b, and so would also have a higher level of overall joint reliability.

Immediately after their deposition, and in the case of electrolytically formed Copper, for some time afterwards, plated Cu layers have a fine nanocrystalline or amorphous structure, and typically undergo significant recrystallisation, which is critical in determining the properties of the final BMV. Through a number of investigations, this paper will identify "bottom-up" and "top-down" recrystallisation, which, respectively lead to a fully epitaxial interface, (Fig 1a) or an interface with significant alignment of Cu-grain boundaries (Fig 1b)



1a 1b

Figure 1.
SEM images showing typical microstructures a) fully epitaxial and b) none epitaxial with clear boundary between the target pad and the via plating

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The onset of, or the prediction of device failure, along with the relative performance of different process conditions is always of interest to designers and engineers alike. However, like many, the PCB the industry has had to rely on extended physical testing in order to investigate this, which due its time constrains, is often frustrating and undesirable. In view of this, we outline a simple inspection methodology by which we aim to quantify the degree of any grain boundary alignment. Referred to as "Normalized Crack Length", we offer this method as a possible means to determine if a plated structure will have a higher propensity to failure when compared to others.

II. Experimental Methods

For the initial investigations into recrystallisation, samples were prepared by depositing 1um of electroless (ELESS) Cu followed by 20um of electrolytic (ELYTC) Cu onto a rolled annealed (RA) Cu foil. Immediately after plating was complete, the sample was immersed in liquid Nitrogen to suspend any further recrystallization and then inspected while kept at cryogenic temperatures. Additional samples were prepared where a thermal exposure of 2hrs at 160°C was performed after plating to ensure full recrystallization occurred, subsequent inspection was performed.

Further test vehicles (TV) for this study were prepared using representative "anylayer" manufacturing techniques and included a "stack" of 8-10 laser formed microvias. The TVs were manufactured by either industrial partners in their facilities, or in an "industry like" Atotech Technical Center. The FR4 dielectrics utilized are commercially available mid T_g (165°C) low loss materials, with BMV dimensions of a nominal 65µm x 65µm. The following general manufacturing operations were applied

- ELESS Cu, 2 variants were considered
    - "Low build" horizontal process with a deposition thickness <0.2um on the target pad.
    - "High build" vertical process with a deposition thickness >0.4um on the target pad
- ELYTC Cu - flash plate 3-5 µm in thickness,
- ELYTC Cu plate – BMV Fill

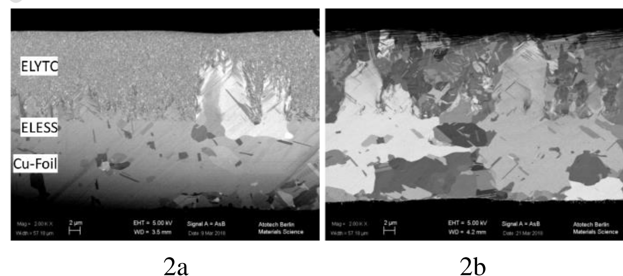
Thermal Cycling Test (TCT) coupons were taken from all TVs, with one group of TCT coupons undergoing multiple hot-oil test cycles [4] (T_{peak}: 260°C for 20 sec) until failure at a resistance increase of >5%. Whereas the second group was stored at ambient conditions without any additional thermal exposure. Columns of stacked vias were randomly selected from both TCT coupon groups and cross sectioned. Investigations were focused on the vias close to the core layer as these typically see higher stress levels.
In order to identify the location of the ELESS Cu layer, each cross section was electropolished and afterwards inspected via SEM (Epol/SEM).

III. Results

<u>"bottom-up" and "top-down" Recrystallization</u>

Figure 2a shows a typical cross section for samples where recrystallization was suspended immediately following plating operations. The plated layer is shown towards the upper part of the image and it is evident that due to the cryogenic treatment, the ELYTC Cu retains its initial but meta-stable, fine grained "amorphous" structure[5]. Towards the lower portion of the plated layers, there is clear evidence that a Cu diffusion based recrystallisation has begun[6], and where this has occurred, the grain structure follows that of the underlaying layer. As such, the structure as a whole is epitaxial to the RA Cu foil and there is no clear delineation across any of the plated interfaces.

In Figure 2b, where the sample has been annealed, we can see that the final Cu structure has now fully developed and again is entirely epitaxial across all interfaces. As the Cu diffusion or "recrystallisation" front is thought to initiate at the Cu foil-ELESS interface, and then proceed vertically towards and through the ELYTC plated layer, it can be described as moving in a "bottom-up" manner, and hence, we refer to this final microstructure as having undergone "bottom-up recrystallisation" For such a structure to occur, it is suggested that all the interfaces between the substrate and each of the plated layers must be sufficiently clean and with a low level of organic contaminants, such that the "recrystallisation front" can pass unhindered through the entire plated structure.



2a 2b

Figure 2.
SEM images showing cryogenically suspended recrystallisation a) immediately after plating and b) after thermal anneal showing "bottom up recrystallisation"

If we now consider the images in Figure 3, where an alternative stabilizer component was used during the ELESS Cu deposition. Immediately after plating (Fig 3a) while the "as plated" amorphous ELYTC Cu is still present, there is no evidence of early epitaxy as seen in Fig 2a, and there is also a clearly identifiable change in the grain structure

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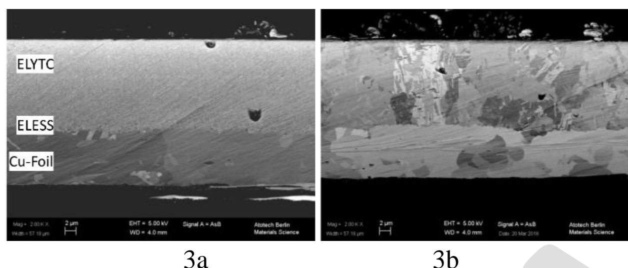
approximate to the location of the ELESS Cu layer. Even after the additional annealing step to facilitate final recrystallisation, the delineation remains, and there is no evidence of significant epitaxy occurring across any of the Cu foil to plated Cu interfaces. In order for such a structure to occur, it is offered that there has been a factor of significance that has reduced the ability of the diffusion front to travel in a "bottom-up" manner, and we subsequently assume that the ELESS and ELYTC Cu have recrystallized in a self-contained manner that is independent to the underlying layer, and so, it could be considered that the overall recrystallisation pattern occurs in a "top-down" mechanism.



3a 3b

Figure 3.
SEM images showing cryogenically suspended recrystallisation a) immediately after plating and b) after thermal anneal showing "top-down recrystallisation"

TVs containing BMVs bearing both epitaxial "bottom-up" and delineated "top-down" microstructures were thermally exposed as described previously to determine if there was any clear relationship between crystal structure and thermo-mechanical performance

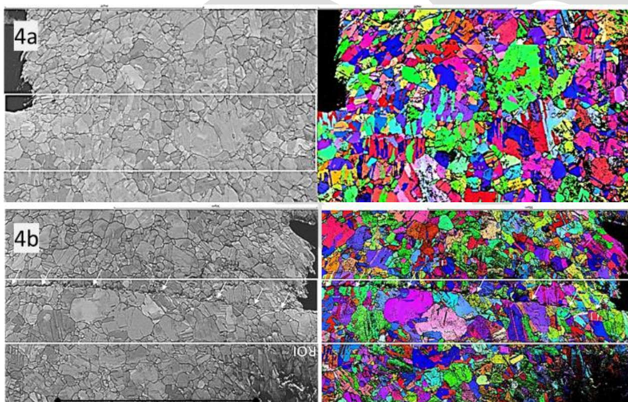


Figure 4.
EBSD band contrast (left) and diffraction (right) images showing micro-structures after thermal exposure typical of a) "bottom up" and b) "top down" recrystallisation in TVs

As expected, there is a discernable relationship between the crystal structure across the BMV interfaces and their respective reliability performance. The "bottom-up" epitaxial structures (Fig 4a) showed no failures during testing, while the "top-down" delineated samples (Fig 4b) had mechanical failure, with cracks propagating close to, or along the point of delineation, which also corresponds to an area of high grain boundary alignment. (Figure 5)

Figure 5.
EBSD diffraction image showing intergranular crack propagation along an area of high grain boundary alignment

If the region of failure is inspected more closely (Figure 6) it is interesting to note that the crack has not only propagated within the initial 500-1000nm of the ELYTC plated layer, but that it also follows the grain boundaries in preference to an epitaxial, but nano voided ELESS-ELYTC Cu interface, which, it would be reasonable to assume, was an area of significant structural weakness. This implies that the cohesion across aligned grain boundaries can be appreciably weaker than epitaxial areas containing significant and aligned nanovoids. So, we could conclude that a BMV containing a fully epitaxial micro structure would offer better reliability performance compared to one with one or more discernable interfaces with significant aligned grain boundaries.

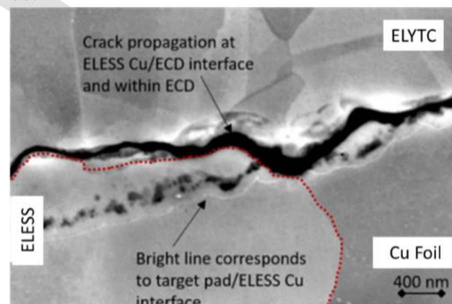


Figure 6.
Electropolished SEM image of the crack path showing failure along aligned grain boundaries in preference to an area of nano voided plated interface.

Recrystallisation Mechanisms

In order for what we describe as "bottom-up" recrystallization to have occurred, it is offered that the amorphous plated layers will be incorporated into the underlying Cu structure through the action of grain boundary diffusion and grain coarsening[6]. In order for this to occur, all interfaces and plated layers must be sufficiently free of contamination so as to not hinder the diffusion process. Figure 7 shows Scanning and Transmission Electron Microscope (SEM and TEM respectively) images of such a joint as well as elemental maps and analysis which have been

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used to determine what may represent a "tolerable level of contamination".

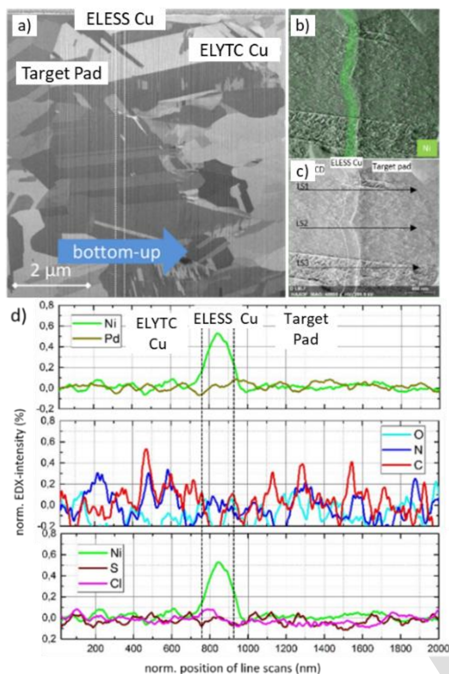

Figure 7.
a) FIB/SEM image of epitaxial interface in a stacked BMV b) TEM image showing ELESS Cu location based on Ni detection c) positions of performed EDX line scans d) averaged EDX intensities of the elemental line scans in c)

The dramatic increase in the Ni content is expected, and should be associated with its use as a refining element common to ELESS Cu deposits. As such, the Ni can be used to determine the location of the ELESS Cu layer within the overall structure. The other elements (C, N, S, Cl), which are typical to either additives in plating electrolytes, or would indicate an organic surface contamination, all remain low and could be considered to be at an acceptable background level, as they clearly have no detrimental effect on the proposed "bottom-up" recrystallisation. The general process of this "bottom-up" recrystallisation is described schematically in Figure 8

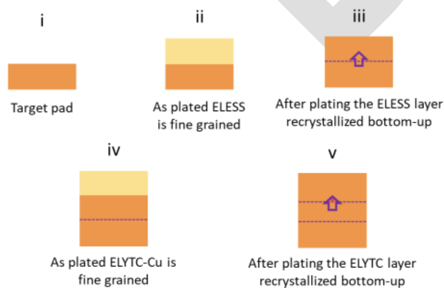

Figure 8
Schematic sequence for "bottom-up" recrystallisation

Where we experience a "top-down" recrystallisation it has been offered that there must be a build-up of contaminants such that the diffusion front is hindered or "pinned" in place which subsequently creates an interface with a high level of grain boundary alignment.

Figure 9 shows the results for the same analysis procedure used previously but in this case with a BMV structure that formed following "top-down" recrystallisation with a visible interface approximate to the ELES Cu layer

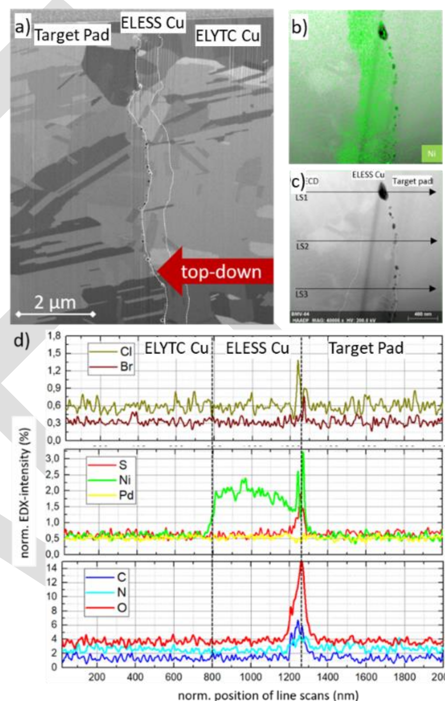

Figure 9.
a) FIB/SEM image of epitaxial interface in a stacked BMV b) TEM image showing ELESS Cu location based on Ni detection c) positions of performed EDX line scans d) averaged EDX intensities of the elemental line scans in c)

Again, using Ni as the indicator for the ELESS layer, its location can be determined, and here it is evident that the ELESS Cu layer has a structure that is epitaxial to the ELYTC Cu, and not to the structure of the underlying Cu foil. This would suggest that movement of the grain boundary diffusion front was retarded and unable to pass from the Cu foil into the ELESS Cu layer, yet was free to do so downwards from the overlying ELYTC Cu.

The EDX line scans, across the same ELESS – ELYTC Cu interface show appreciable increases for a number of elements (Ni, Cl, Br, S, C, N, O) giving a strong indication of a high level of contamination of the target pad surface prior to ELESS Cu deposition. In this case, the Br is taken to be associated with the epoxy resin system and indicates

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inadequate cleaning. While the unusually high Ni, S, Cl & N content suggest a level of ELESS liquid entrapment associated with the resin residues.

Here we can conclude that an appreciable level of surface contamination, from desmear and rinse water resides were present on the target pad, and were of sufficient concentration to hinder grain boundary diffusion from the Cu foil into the ELESS Cu layer and the diffusion front was suspended at this interface. Subsequently the recrystallisation of the ELESS and ELYTC Cu occurred independently of the target pad, and so can be described as "top-down" in manner.

Figure 10 again shows the schematic of the sequence for the contaminates at the target pad surface impacting the diffusion and subsequent recrystallisation

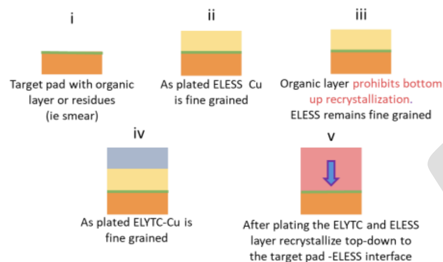

Figure 10
Schematic sequence for "bottom-up" recrystallisation

Clearly, situations will likely arise where combinations of the above two mechanisms could occur. For example, If the target pad – ELESS interface is clear of contaminants yet the ELESS – ELYTC Cu interface is contaminated, due to a heavy incorporation of ELYTC plating additives for example, then it would be reasonable to assume that the point where any grain boundary alignment would occur, would shift up to a location approximate to the ELESS - ELYTC Cu interface. As such, the ELESS Cu would "bottom-up" recrystallize while the ELYTC Cu would likely follow a "top-down" mechanism.

<ins>Normalized Crack Length as an Indication of Reliability</ins>

While it is indeed interesting to appreciate the above approach with respect to extreme crystal structures, how they form and the interdependency they have with mechanical performance, it would also be beneficial if there was a numerical approach that could be applied to determine which of two similar looking crystal structures would have a greater propensity to failure. "Normalized Crack Length" (NCL) is offered as a means to enable this as it makes use of the crystallographic features within a BMV as a means to determine a potential crack path, and the longer and more tortuous that path, the greater the likely of improved reliability performance, i.e. a high NCL is beneficial.

In simple terms the NCL is the ratio of a predicted or "tentative" crack length in comparison to the actual plated interface length. The outline process is described below, and an example is shown in Figure 11.

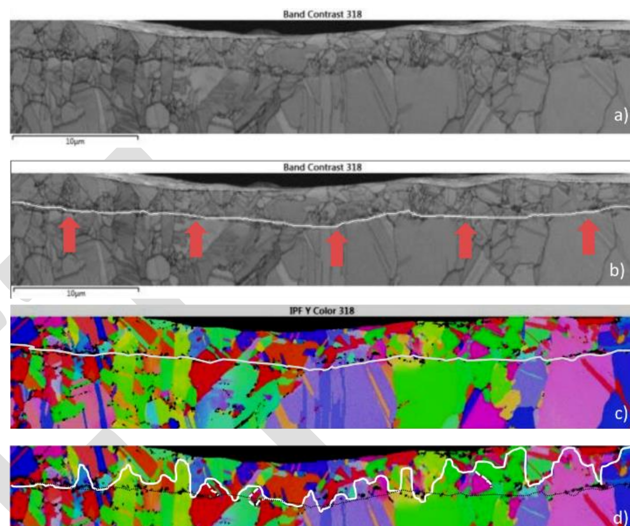

Figure 11
Steps for determining Normalized Crack Length
a) identify Interface Location (IL). b) mark and measure IL
c) Overlay onto EBSD diffraction image d) identify and measure Tentative Crack Length (TCL)

1. Cross section and obtain a clear image centered on the target pad – ELESS – ELYTC interface. This may be best achieved through electro polishing, or band contrast imaging (Fig 11a) At the same time, take an EBSD diffraction image as this will be needed later.
2. Mark a line that follows the observable interface and using suitable graphic software, measure and assign a length to the observed line. This value is referred to as the "Interface Length" (IL) (Fig 11b)
3. Overlay the location of the IL onto the EBSD diffraction image taken previously (Fig 11c)
4. Starting at one side of the image, begin to determine the most likely crack path that is approximate to the identified interface in step 3 (Fig 11d) The following is recommended when determining the most likely crack path
    - The crack will always travel and continue to travel in the forward direction ie, it will not "double back" on itself
    - The crack will always follow grain boundaries
    - The crack will always take the shortest possible route

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- The crack will always continue to propagate, i.e. it will not self-limit and stop

5. Using the graphic software determine and assign a length to the identified crack path. This value referred to as the "Tentative Crack Length" (TCL)
6. Calculate Normalized Crack Length (NCL) using the following equation

$$NCL = \frac{Tentative\ Crack\ Length\ (TCL)}{Interface\ Length\ (IL)} \quad (1)$$

In the example shown in Figure 11. The IL and TCL were determined to be 52µm and 82 µm respectively, with the calculated NCL being 1.6.

In terms of identifying an ideal or desirable NCL value, this is still under investigation and should be considered a "work in progress" However, it is clear that a value of 1.0 would represent a short crack that would directly, or very closely follow the plated interface, and as such would describe a joint showing a high level of grain boundary alignment, and as discussed previously would be expected to be mechanically weak. An NCL of 2.0 or higher is considered to represent a fully epitaxial grain structure that is typical to the via fill plating, and so would be expected to offer the best opportunity for higher mechanical performance. Typically, it has been found that calculated NCL values lie between these two extremes, and while this is still under investigation, it is thought that any target, or desirable NCL value would likely lie approximately mid-way.
However, it is vital to consider that as TCL is determined manually, it is questionable if one can directly compare NCL values when they are offered from different sources. It is reasonable to assume that if 2 users interpret the same sample, there will certainly be differences between their approach in deciding the crack propagation path, and TCL. As such user 1 may calculate a final NCL of 1.54 while user 2 could easily conclude it to be 1.24 or even 1.84. In view of this it would be interesting to consider if the calculation of NCL could be performed automatically through the use of Artificial Intelligence or other approaches which could remove any "human error"

IV. Conclusion

The crystal structure in plated BMVs is a critical factor which can be directly linked to their mechanical performance, however there has been little direct work investigating or attempting to explain how they form. In this paper, we offer the concepts and mechanisms for the two major types of observed strictures. "bottom-up" recrystallisation is considered to yield an epitaxial structure which is considered optimal for product reliability and will form in the absence of significant contaminants across the plated interfaces. Meanwhile, "top-down" recrystallization occurs when contaminants are present at sufficient level to impede the necessary grain boundary diffusion and will result in a weakened joint which will subsequently exhibit a high level of grain boundary alignment.
The concept of a Normalized Crack Length has been introduced and offered as a potential means to evaluate plated BMVs with a view to determining their relative propensity to the onset of failure. While investigations using the method itself have shown definite merits, it should also be considered that the technique is still in its infancy and will require further development to minimize user interpretation which may lead to inconsistencies.

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