Microstructural and Orientation Studies of Copper Crystals

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Abstract

With the advance of technology in consumer electronic industries, there is an urge in miniaturization, environmental friendly and functionality enhancement of the electronic products. This requires increasing the wiring density of electrical active layers in the printed circuit board (PCB) and the ability to stand higher lead-free reflow temperature. These impose stability and reliability issues on the copper interconnection structures, especially at the through-hole and via regions. In order to minimize the electrical connection failures in the PCBs, it is of vital importance to explore a method suitable to understand and identify the origin of failure. While it is accepted that grain size, orientation and its distribution show remarkable impacts on the physical and mechanical properties of the copper wiring lines, electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) seem to be suitable as the analytical techniques. In this paper, a scanning electron microscope equipped with EBSD was employed to investigate the grain structures of copper. The results demonstrated that EBSD is a potential analytical candidate in getting insights into the reliability issues of copper interconnects.

Introduction

With the advance of technology in consumer electronic industries, there is an urge in miniaturization, environmental friendly and functionality enhancement of the electronic products. This requires increasing the wiring density of electrical active layers in the printed circuit board (PCB) and the ability to withstand higher reflow temperature for lead-free technology. These impose stability and reliability issues on the copper (Cu) interconnection structures, especially at the through-hole and blind-via regions. In order to minimize the electrical connection failures in the PCBs, it is of vital importance to explore a method suitable to understand and identify the origin of failure.

Chemical etching is a direct, quick and simple metallographic method for investigating the microstructure of electrolytic Cu in PCBs. By performing plain-view and micro-sectioning of failed PCB sample, the presence of cracks, abnormal grain growth and trapping of gas bobbles could be easily revealed. It is understandable that cracks found in the electrolytic Cu layer normally initiates at stress concentration regions such as corners of through-hole and blind via. The propagation of cracks would be facilitated by the presence of columnar grain structures where the presence of highly directional grain boundary could not act as an effective crack arrester. Thus, a fine and equaxial grain structure is mechanically preferred to prevent from electrical connection failure. However, the grain structure of electrolytic Cu in PCBs sometimes looks normal just after electroplating. Failures of PCBs, in reality, occur after assembling of electronic components or operating at extreme ambient condition. As a result, it would be preferable if the grain structure could be tracked after electroplating. A structural correlation between the initial normal-and final failed grain-structure can be constructed. Ultimately, a special grain structure could be developed to suppress the initial of cracks and to arrest any propagating cracks through grain boundary engineering.

Characterization techniques appropriate for grain analysis

Corresponding micro-structural characterization techniques such X-ray diffraction (XRD), electron

	XRD	EBSD	TEM
Sample constraints	Low/Non-destructive	Medium/ Destructive	High/Destructive
Information obtained	Bulk (averaged) Through-thickness, > µm	Local Surface, ~ hundreds of nm	Local Through-thickness, < µm
Crystal size	✓	✓	✓
Crystal orientation	✓	✓	✓
Distribution of grain size & orientation	×	✓	✓
Texture analysis	✓	✓	✓
Grain-to-grain misorientation	×	✓	✓
Imaging	×	✓	✓
Labor intensitve	Low	Medium	High
Cost	Low	Medium	High

Table 1. Comparisons of XRD, EBSD and TEM for grain analysis

backscatted diffraction (EBSD) and transmission electron microscopy (TEM) and even focused ion beam (FIB) microscopy have been developed for determination of grain size and texture component. Each characterization technique has its own advantages over the others and some of them may be complementary to each other. A brief comparison of XRD, EBSD and TEM for grain analysis has summarized in Table 1. XRD is a non-destructive and simple method capable of obtaining the size and orientation of grains in addition to texture analysis. However, the signal acquired is bulk averaged with a probing depth in micrometer range under theta-two theta acquisition mode. It is widely recognized that TEM features the highest level of information necessary for micro-structural analysis of grain, even capable of providing resolution down to atomic level. However, the operating cost is rather high and the sample preparation is tedious. As a result, this imposes the infeasibility in performing statistical analysis on large amount data. On the whole, it seems that EBSD is a much appropriate technique balancing the advantages and disadvantages of XRD and TEM. This is attributed to the active development of high speed charged-couple device (CCD) camera technology and software development of algorithmic transformation that substantially reduces the acquisition and analyzing time from tens of hours to tens of minutes since its early stage of development from the academic institute.

An electron backscattered diffraction (EBSD) detector as depicted in Fig. 1 is an auxiliary primary backscattered electron detector in addition to a secondary electron detector (SED), an energy dispersive X-ray (EDX) spectrometer, and a classical semiconductor type backscattered electron detector equipped in a typical SEM chamber. In order to maximize the signal of forward scattered electrons arising from the interaction of energetic electrons with the surface layer of a specimen, the sample surface is tilted at 60 to 70 degree towards the phosphor screen. A CCD video camera with inherently high sensitivity to a color range is installed at the back of phosphor screen in order to record down the electron backscattered diffraction pattern. The diffraction pattern is basically composed of a series of Kikuchi bands (Fig. 1c), which confines characteristic information about the crystallographic plane of a sample under examination. If an energetic electron beam is raster-scanned over an area of interest, their corresponding grain structures could be obtained by assigning a colour to each diffraction pattern. Similar diffraction pattern will result in same colour designation. By transforming all the data points to its colour counterpart, a grain color image map could be obtained.

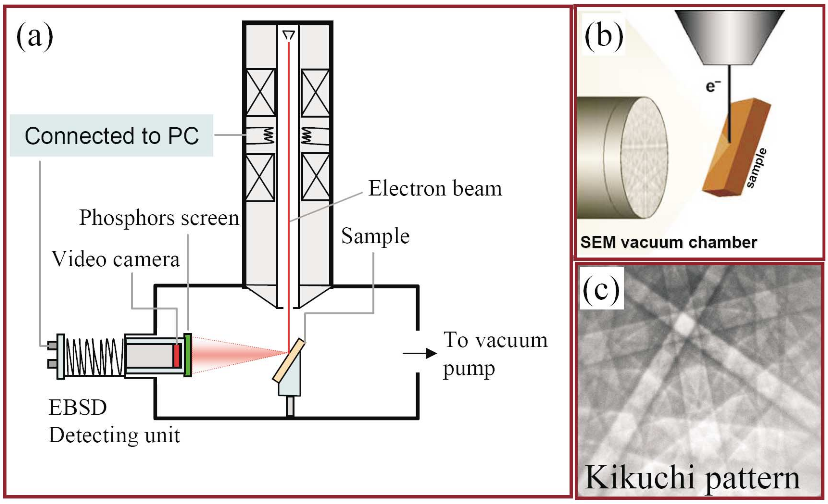

Fig. 1 (a) Schematic showing an electron backscattered diffraction detecter equipped in an SEM chamber. Enlarged region (b) elucidating the interaction of electron beam with sample surface and (c) formation of a corresponding Kikuchi pattern.

General information obtained by electron backscattered diffraction analysis

Fig. 2 illustrates some typical data (a) grain orientation image map, (b) inverse pole figure and (c) pole figure obtained from a [111] oriented bi-crystal Cu sample by EBSD analysis. The low index planes (100), (110) and (111) are represented by red, green and blue respectively as shown in the triangle colour legend. The bi-crystal Cu sample was intentionally employed as a standard sample elucidating how the EBSD working for grain micro-structural analysis. The analyzed region was closed to the interface between two Cu (111) crystals deviated by several degrees. This interface leads to the formation of a small angle grain boundary. Obviously, only blue/purple colouration was revealed, which is an indication Cu {111}planes according to triangle colour legend. By performing a statistical summary of the crystallographic planes on the analyzed region, an inverse pole figure (IPF) map (Fig. 1b) can be obtained. Since only two discrete spots were observed, this implies the presence of two sets of Cu (111) planes deviating by a small angle. Pole figure (PF) map is especially useful when performing texture analysis on samples having in-plane texture. For the bi-crystal Cu sample studied in this paper, the PF map (Fig. 1c) is relatively simple. The central point as indicated by a cross-hair in the PF map is assigned as 0°. The circumference in horizontal and radial direction is designated as 90°. The appearance of a dot closed to the cross-hair indicates a preferential [111] orientation of Cu crystal was formed. Regular discrete dots were found at about 70°, which is attributed to second set of Cu (111) plane as the intercept between two Cu (111) planes is 70.5°. Since a continuous ring is absent, an in-plane texture is not observed for bi-crystal Cu sample.

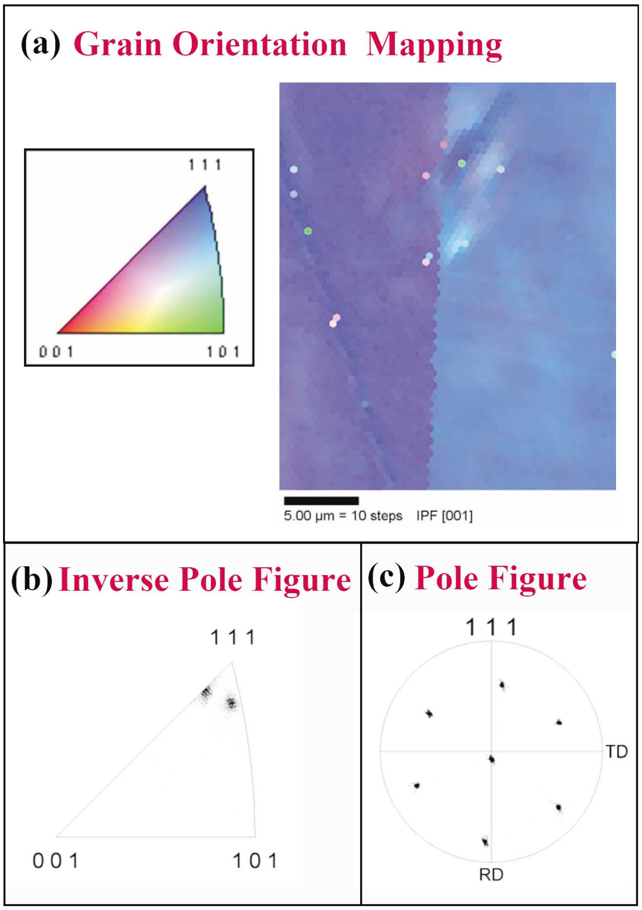

Fig. 2 Typical data (a) grain orientation mapping, (b) inverse pole figure and (c) pole figure obtained from a [111] oriented bi-crystal Cu sample by EBSD analysis.

Electron backscatter diffraction analysis of electroplated Cu on PCBs

After having a basic concept about the EBSD analysis of a known standard Cu sample, subsequent session will attempt to illustrate the application of EBSD analysis to electroplated Cu in planar and cross-sectional view. The electroplated Cu under this investigation was deposited using acid copper plating with the presence of proprietary organic additives. This paper will not discuss in detail about the influence of organic additives on the resulting grain structure. Instead, the capability of the EBSD applied in electroplated Cu will be exploited. Fig. 3 displays a typical electroplated Cu sample probing at plane-view: (a) an SEM topographic image revealing a featureless surface morphology, (b) a crystal orientation map acquired by EBSD showing randomly orientated micrometer-sized grains and (c) IPF and PF maps indicating no sign of preferred texture formation. Since the black dots are distributed randomly over the triangular map, this implies that there was no

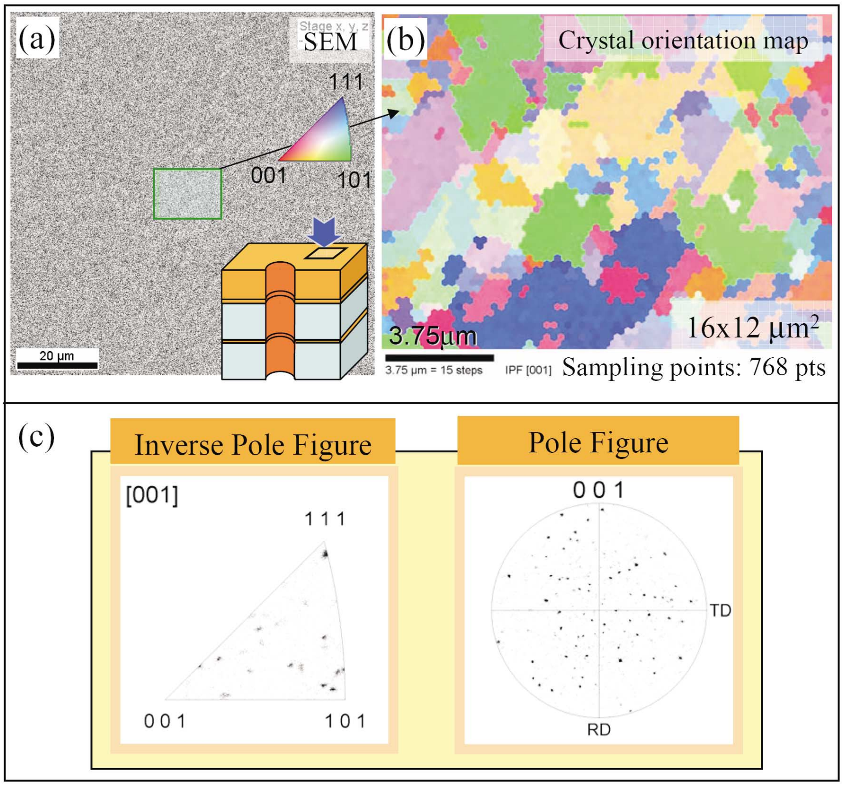

Fig. 3 A typical electroplated Cu probing at plane-view: (a) SEM topography revealing a featureless surface, (b) crystal orientation map acquired by EBSD showing randomly orientated micrometer-sized grains and (c) IPF and PF maps indicating no sign of preferred texture formation.

preferred crystallographic orientation for the electroplated Cu sample. Similar results were also obtained in the PF map due to the absence of regular discrete spots or continuous rings such map. For direct grain analysis on an internal surface inside the through-hole, it is nearly impossible to acquire an indexable EBSD image. This is attributed to inherently high internal surface roughness inside the through-hole, which will ruin the formation of Kikuchi patterns due to geometric shadowing of electrons backscattered from the analyzing sample surface. Thus, a cautious sample preparation step (Fig. 4) must be followed in order to get consistent result. Prior introducing the mechanically polished sample into the SEM chamber for EBSD analysis, it is recommended to perform wet chemical etching and vacuum argon ion sputtering in sequence in order to reduce the surface deformed layer and surface oxidized layer. For sample prone to oxidation at ambient environment, a thin amorphous carbon layer is required to prevent the clean surface from further oxidation.

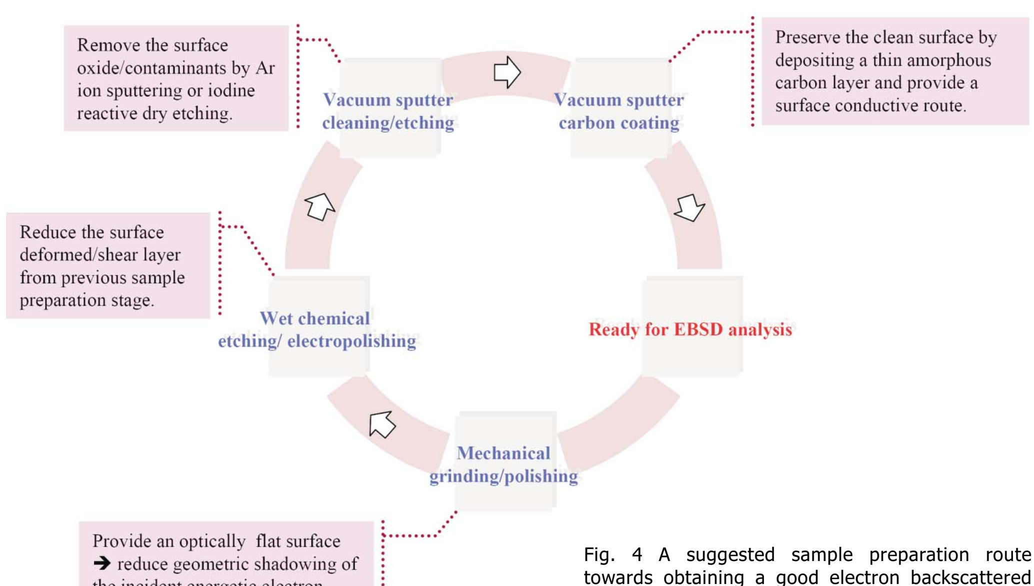

Fig. 4 A suggested sample preparation route towards obtaining a good electron backscattered diffraction pattern.

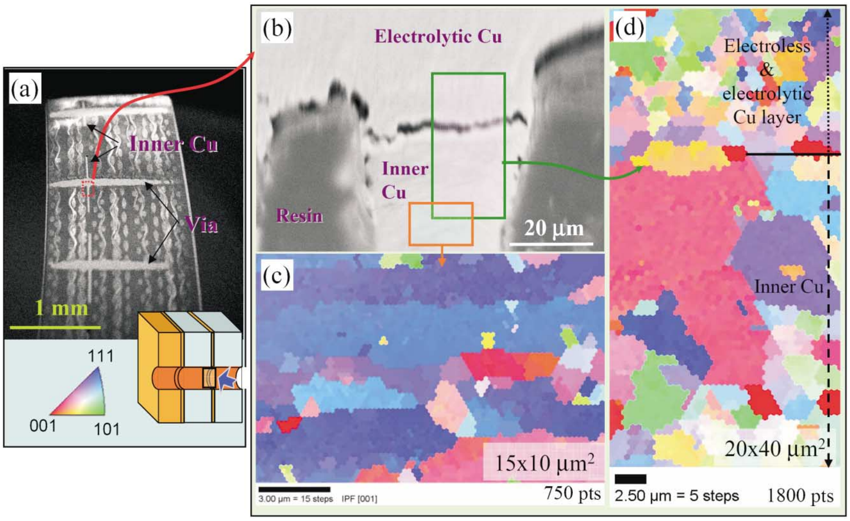

Fig. 5 A typical electroplated Cu in cross-sectional view: (a) an SEM micrograph revealing the inner Cu and metallization layer; (b) a magnified image showing the connection between the inner and electrolytic Cu; (c) a crystal orientation image map of inner Cu and (d) interface between electrolytic and inner Cu.

Fig. 5 displays a typical electroplated Cu in cross-sectional view: (a) an SEM micrograph revealing inner Cu and metallization layers; (b) a magnified image showing the connection between the inner and electrolytic Cu; (c) crystal orientation image map of an inner Cu and (d) an interface between electrolytic and inner Cu. The images shown in Fig. 5 are obtained by preparing the micro-section closed to the tangent of through-hole. Since the overall thickness of Cu is typically less than 20µm, special attention has to draw during the course of mechanical grinding/polishing. Otherwise, the metal Cu layer will be removed easily. It is observed that highly-oriented grain structure dominating by columnar structure with [111] orientation was found for the inner Cu layer. Their crystal size was relatively larger as compared to the electrolytic Cu. Such type of elongated grain was not observed for the electrolytically plated Cu no matter it is closed to or far away from the interface. The overall crystal size of electrolytic Cu increases weakly with coating thickness as elucidated in Fig. 6. The orientation of Cu crystal remained randomly oriented with increasing coating thickness.

Concluding remarks

By getting more insight into the grain structures of electrolytic Cu in PCBs by a novel characterization technique like EBSD, future generation of electrolytic Cu can be tailored with special grain structures that can operate at harsh environment with less concern about the reliability issue.

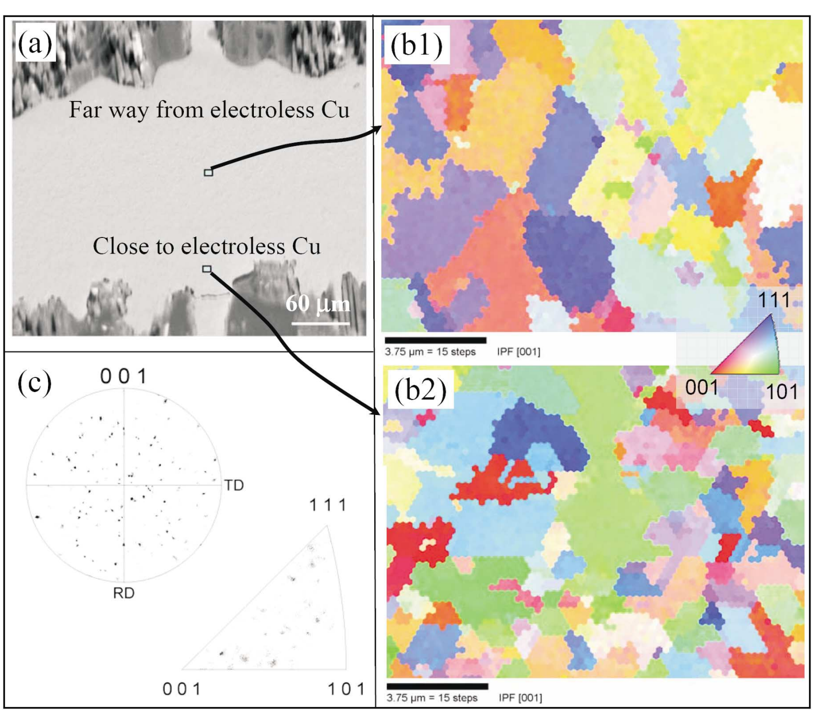

Fig. 6 Comparison of grain structure between the region (b1) close to and (b2) far away from (b2) electroless Cu. Their grain structures are randomly oriented as retrieved from (b).