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Fracture of micrometre-sized Ni/Au coated polymer particles

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Abstract
Deformation and fracture of individual micrometre-sized Ni/Au coated polymer particles have been studied by a nanoindentation-based flat punch method. A wide range of test conditions has been applied to deform the coated and the uncoated particles. The compression induced cracking of the Ni/Au coating and delamination between the metal coating and the polymer core have been investigated and provide insight into the effect of nanoscale metal coating on the deformation capacity and fracture process of the particles. A three-stage deformation and fracture behaviour of the particles have been identified. The results are essential for the design of metal coated polymer particles for industrial applications.

1. Introduction
The Ugelstad method is a well-known and versatile technology for manufacturing highly monodisperse polymer particles [1]. The technology has been proven highly successful within chemical and biological industries [2]. In the past decade this technology has been exploited for use within electronics and microsystems. By metallizing polymer particles they can be utilized as flexible electrical contacts in numerous potential applications [3]. Until now, the focus has been on the use of metal coated polymer particles for developing new electrical packaging technology, such as anisotropic conductive adhesive (ACA). Usually the metal coated polymer particles used in ACA application have a polymer core—double metal layer structure that consists of a polymer core for improving contact compliance, Ni inner layer for electrical conductivity and Au outer layer for protecting the inner layer from oxidation and increasing the reliability of electrical performance. Therefore, the ACA technology is of high interest because it is lead-free, reduces package size and achieves high-density interconnections. During the ACA bonding process, the electrical characteristics are strongly connected to the contact area between the chips and the metal coated particles. A large contact area is required to enhance the electrical performance as well as the reliability of the interconnection. Therefore, a large deformation is applied to the metal coated particles to obtain a reliable and low resistance connection, which may result in the failure of the metal coated particles. This can create a significant impact on the electrical performance of the interconnect. For these reasons a detailed knowledge of the mechanical properties of individual particles is essential for the design of electrical assemblies.

However, only limited research has been conducted regarding the mechanical properties of the micrometre-sized polymer particles because of the small volume and spherical geometry involved. The aggregate mechanical properties of a large number of particles (typically several hundred) have been previously estimated through grouping particles between two polished silicon chips [4]. For individual particles, a nanoindentation-based flat punch method has been recently developed to measure the mechanical response of both metal coated particles and uncoated polymer particles [5–9]. The contact load–displacement relationship, large deformation and size effect of individual particles have been investigated. However, the study of deformation and fracture processes on metal coated polymer particles has not been reported to date. The goal of this work is to characterize the deformation behaviour and fracture track of metal coated polymer particles.
2. Experimental setup

The metal coated polymer particles used in this work were 3.8 µm in diameter consisting of a strongly crosslinked acrylic copolymer core coated with Ni and Au layers, as shown in figure 1(a). The coefficient of variance (CV) of the particle size distribution is 1.7%, where CV is defined as the ratio of the standard deviation to the mean diameter. The polymer core is amorphous and has been synthesized by using the Ugelstad method with a multi-step swelling process [1]. The Ni/Au coating was plated on the polymer particle surface with a Ni inner layer of about 50 nm thickness and a Au outer layer of about 25 nm. For comparison, uncoated particles with the same polymer chemistry and the same size were also prepared and tested.

The mechanical test was performed using a commercially available nanoindentation device (TriboIndenter® Hysitron Inc., MN, USA). The nanoindentation-based flat punch method was employed to characterize individual particles, schematically shown in figure 1(b). A diamond flat punch of 100 µm diameter was specially designed to compress the particles instead of a common sharp tip used for nanohardness measurement [10–12]. The planarity and the parallelism of the flat punch are significant to the test precision and have been carefully calibrated through the impression on the well-polished indium surface. The standard load-control mode in which the applied force on the specimens follows a predefined load function has been used. A three-step loading protocol has been set, which contains linear loading/unloading segments with a 2000 µN s⁻¹ loading rate.

3. Results and discussion

Typical compression force–deformation curves for a Ni/Au coated polymer particle and an uncoated polymer particle are shown in figure 2. Here deformation is defined as the ratio of the deformed particle height to the undeformed diameter. The loading behaviour of the coated particle can be divided into three stages compared with the loading behaviour of the uncoated one. In stage I, the stiffness of the coated particle is prominently higher than the uncoated one and increases monotonically until about 18% deformation. In stage II, the deformation increases from 18% to around 43%. A displacement burst at about 18% deformation indicates that a very significant event is taking place in the Ni/Au coating, probably including both cracking and delamination from the polymer core. Within the 20 ms timeframe of the displacement burst, given by the measurement sampling frequency, the deformation rapidly increases from typically 18% to 23%. However, the coated particle behaves still harder than the uncoated one, indicating that there are some mechanical integrity in the metal layer and adhesion to the polymer core left. Once the deformation exceeds around 43%, the loading curves of the coated and uncoated particles seem to overlap each other and the metal coating does not influence the particle behaviour in stage III. The results reveal that the Ni/Au coating initially plays a significant strengthening effect on the mechanical behaviour of the particle. As the deformation is up

Figure 2. Representative compression force–deformation curves of a metal coated and an uncoated polymer particle.

A tiny amount of particles were dispersed onto a polished silicon chip of the size 10 x 10 x 0.5 mm³. Using the integrated optical microscope, individual particles with more than 75 µm distance to the closest neighbour were identified for the test. The chosen particles were tested only once, which means that each test was performed on a virgin particle. For each set of experiments, at least five individual particles were tested in order to check the repeatability of the results. After the mechanical test, the morphological observation of the tested particles was performed using a scanning electron microscope (SEM) Zeiss Ultra 55 LE FESEM.
to approximately 18%, the influence of the coating gradually decreases. Until approximately 43% deformation where the effect of the Ni/Au coating disappears completely, the coated particle behaves the same as the uncoated particle.

Figure 3 shows the representative force–deformation behaviour of Ni/Au coated particles compressed to five applied peak loads 1000, 1500, 2000, 3000 and 10 000 µN with loading/unloading rate 2000 µN s\(^{-1}\). The loading segments of the five curves, and even the displacement burst points at approximately 18% deformation, are remarkably coincident. The characteristic SEM images of the corresponding five particles are shown in figure 4. All images are taken from the top view, in the direction of the compression.

The loading behaviour of the particle tested at 1000 µN peak load is located in stage I. The maximum deformation of the particle is about 11% and less than the displacement burst deformation 18%. After unloading the particle recovers completely without any residual deformation. The hysteretic effect observed in the force–deformation curves is mainly caused by the viscoelastic properties of the polymer core [13, 14]. The corresponding SEM image of this particle is displayed in figure 4(a). The microcracks can be observed within what has been the contact area between the flat punch and the particle. Because of the complete recovery, these microcracks can be considered as the local response to the initial deformation and have a negligible influence on the macroscopic behaviour of the particle.

Increasing the peak load to 1500, 2000 and 3000 µN, the loading behaviours of the particles go into stage II in figure 2 and the force–deformation curve passes through the burst point. In these cases, the particles partly recover and have a residual deformation after unloading. The micrographs of three particles are shown in figures 4(b)–(d). A flattened surface area in each image is observed which indicates residual deformation and corresponds to the expected contact area under maximum deformation. All images clearly exhibit cracking of the Ni/Au coating and delamination between the metal coating and the polymer core. The cracking and delamination are obviously aggravated with increasing peak loads. The crack propagation is parallel to the longitudinal direction of the particles, and the delamination usually occurs underneath the cracks or by the warping of the Ni/Au coating. In figure 4(d) two microcracks on the top-left are observed, probably starting from the ‘bottom’ side of the particle. The results demonstrate that the displacement burst in stage II is caused by the cracking of the Ni/Au coating or the delamination between the Ni/Au coating and the polymer core or a combination of the two. From there on the strengthening effect of the metal coating is reduced dramatically.

The final fracture of the entire particle is observed in figure 3, which means the polymer core is also crushed and presented by the second displacement burst at about 58% deformation on the force–deformation curve. The crushed fragment of the particle is shown in figure 4(e). The micrograph explicitly shows the polymer core fracture together with cracking and delamination of the Ni/Au coating. It is difficult to identify the fracture direction of the polymer core because the particle is possibly rotated during the final fracture. By comparing two force–deformation curves presented by the solid square lines in figures 2 and 3, it is worth noting that not all coated particles show the polymer core fracture. A statistical result that about 50% coated particles showed the final fracture has been obtained by additionally compressing a number of individual particles. However, there was no fracture observed on the uncoated particles at the same 10 000 µN peak load. There are three possible explanations for this observation. One is that the Ni/Au coating fragments might induce defects to the polymer core during the cracking of the coating. As shown in figure 4(e), the coating fragment marked by an ellipse has entered the polymer core and possibly creates an initiation site for the polymer core fracture. The second explanation is that the total load carried by the Ni/Au coating is transferred to the polymer core instantly when the cracking of the coating happens. This may induce an impact effect to the polymer core. Considering that the coated particle behaves almost identically with the uncoated particles when deformation is up to 43%, that is to say, no significant dynamic effect is observed on the coated particle in this region. Therefore, this ‘impact’ mechanism is of secondary nature. The last explanation is that the polymer core could be lightly altered during the plating process. A typical plating process involves a relatively strong etching on the polymer surface to introduce nucleation sites.

4. Conclusions

This work investigates the effect of the metal coating, and its cracking and delamination on the deformation behaviour of the Ni/Au coated polymer particles. According to the effect of the metal coating, three stages could be identified in the deformation process. Initially the Ni/Au coating has a striking strengthening effect, where the deformation is contributed mainly by stretching the Ni/Au coating so the coated particle is harder than the uncoated one. Secondly, the effect of Ni/Au coating is significantly reduced when the cracking of the Ni/Au coating and the delamination between the Ni/Au coating is...
coating and the polymer core occur. The critical deformation for the cracking and delamination of the metal coating is around 18%. In the third and last phase with a deformation above approximately 43%, the coated particle shows nearly identical behaviour as the uncoated one. Fracture of the polymer core does partly occur on metal coated particles while not on uncoated polymer particles. These results are integrated effects of particles geometry and material. The findings have important implications in the design of the metal coated polymer particles for the ACA application.

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References