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Thermal and Mechanical Effects on Silicone (De) Intercalation in Pressure Sensitive Adhesive

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***Abstract*— *The adhesion layer integrity of a polyamide-polyacrylate system as a function of thermal and mechanical treatment are explored by looking into the interaction/de-intercalation. Thermal treatment induced the intercalation of silicone into the adhesive layer, which is affected by the temperature and duration of the treatment. The mechanical treatment resulted in the de-intercalation of the silicone strands. These results suggest the mobility of the silicone strands affects the integrity of the polyamide-polyacrylate adhesion joint, and the preferential interaction of the silicone to the polyamide surface at room temperature and to the polyacrylate adhesive at elevated temperature.*

***Keywords*— *Adhesion, polyacrylate, polyamide, silicone, TOF-SIMS.*

I. Introduction

The adhesion between materials is an important concept in many industrial applications such as in the microelectronics, biomedical, automotive and aerospace industries. The adhesive joint formation and its integrity determine the reliability of the resulting product [1].

The polyamide-polyacrylate adhesive joint is indispensable in the microelectronics industries [2], often utilized in molded or cavity packages. Polyamide substrates requiring functional or cosmetic protection against external stimuli use liners comprising of a base material, a thin adhesive film, and a cover layer. Conformable polyacrylates are among the materials of choice due to their tunable properties [3-4]. Residual impurities such as silicone, used as release agent in liners, could disrupt the polyamide-polyacrylate joints when substantial amount is trapped with the adhesive interface [5].

Silicone-based materials are used as the release agent to facilitate easy peel off prior to application. Optimization of the amount of silicone is critical in designing the functionality of the material, controlling the tack (or lack of it). In cases where there is very low concentration of silicone, hard to pick issue is encountered. Contrary, too much silicone results in non-stick issues. If applied to another surface, the amount of silicone is one of the critical factors dictating the adhesion integrity and reliability. Preferential interaction of silicone with the adhesive layer or the other surface further complicates the adhesion strength [6].

Herein, we explore the thermal and mechanical effects, encountered during the functional life of the pressure sensitive adhesive (PSA), on the silicone interaction/de-intercalation. TOF-SIMS spectral data provided some mechanistic insights into the motion of the silicone strands as a function of thermal and mechanical events applied on the system.

II. Experimental Details

3M and Kuraray manufactured the polyacrylate-based pressure sensitive adhesive (PSA, 7418S) and the substrate's polyamide material (Genestar PA9T), respectively. The polyacrylate-polyamide adhesion interface (Fig. 1) was created by attaching the adhesive to the substrate using a jig with a 200 gf press at 50 s dwell time. The adhesive joint was subjected to a 7 h dry bake at 125 °C (Thermal 1), 6x drop test at 4 ft height (Mechanical) and 3x reflow at 250 °C peak temperature (Thermal 2). The fresh substrate, the cleaved adhesive-substrate surface after attach, and after thermal and mechanical treatment were subjected to mass spectral analyses. The ToF-SIMS analyses were performed using IONTOF TOF.SIMS5, equipped with a Reflectron TOF Analyser and EDR Secondary Ion Detector, using Bi+ primary beam on a 200 μm x 200 μm surface.

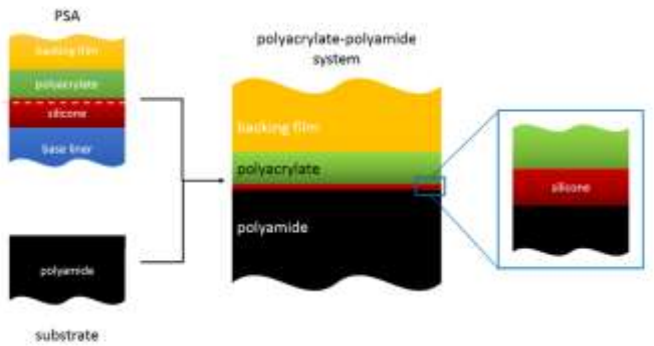

Fig. 1. Build-up of the adhesion interface, from polyacrylate-based PSA and polyamide substrate, with residual silicone [6].

III. Results and Discussion

The nature and integrity of the interfacial interaction between the polyamide substrate and the polyacrylate PSA, in the presence of residual silicone, forming an adhesive joint are evaluated by forming the interface as illustrated in Fig. 1. The silicone is present as a release agent to separate the backing film with the adhesive layer from the base liner. The cleaved material is attached with the substrate with any residual silicone expected to be present within the interface. The effect of the thermal and mechanical treatment on the mobility of the silicone strands was elucidated by measuring the surface availability of the silicone after each treatment.

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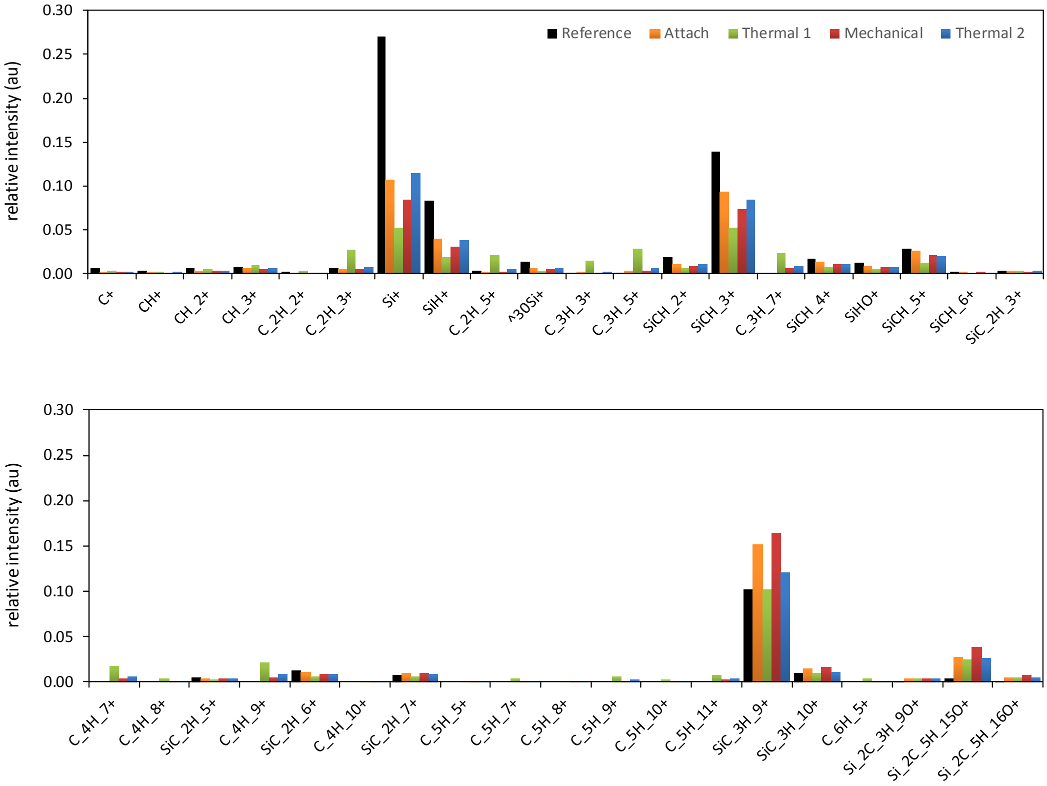

Fig. 2. TOF-SIMS positive scan molecular fragments from the polyamide surface at different process conditions.

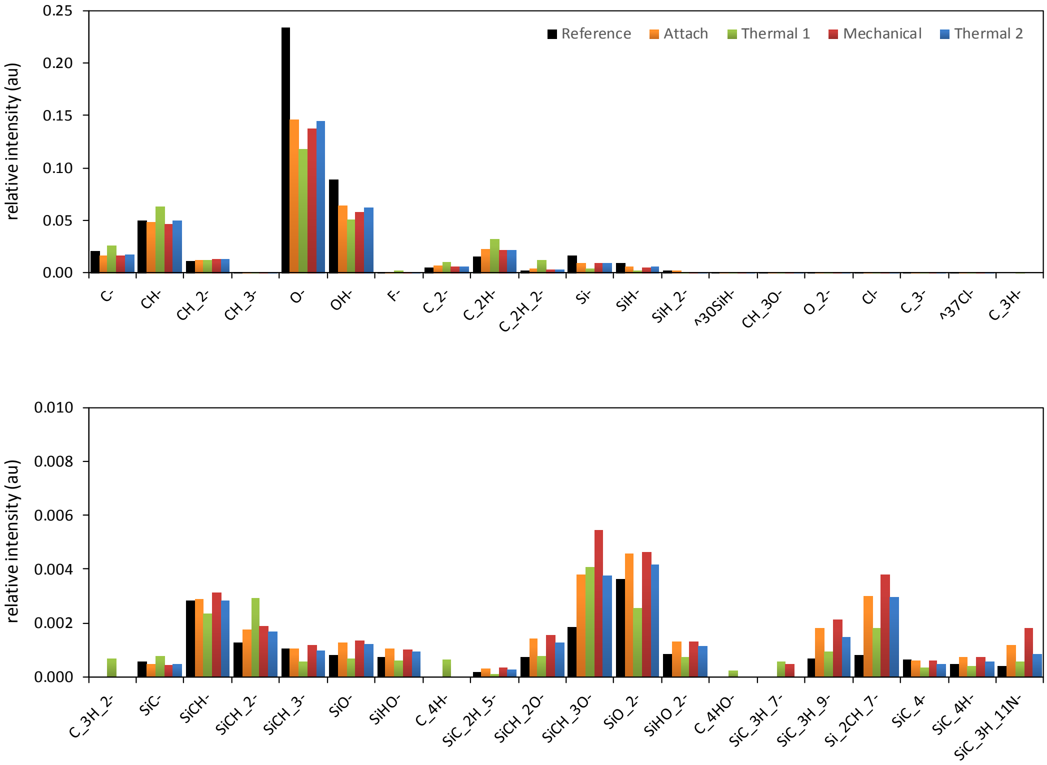

Fig. 3. TOF-SIMS negative scan molecular fragments from the polyamide surface at different process conditions.

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TOF-SIMS spectral analyses were performed on staged PSA liners to assess the effect of staging on the surface components of the liners. Fig 2 shows the positive spectral molecular fragments indicating the presence of two significant groups, the alkyl fragments and the silicon-containing fragments. Similarly, Fig. 3 reflects the existence of these two functional moieties. The alkyl fragments primarily originate from the adhesive layer; hence, it was used as the surface determinant for the adhesive layer. Similarly, the silicon-containing fragments confirm the presence of the residual silicone. It is interesting to note that the polyamide surface is dominated by the residual silicone, originally coming from the PSA. This indicate the preferential interaction of the silicone strands with the polyamide surface versus the polyacrylate adhesive layer at room temperature.

The effects of the thermal and mechanical treatments experienced by the adhesion joint were determined by analyzing the silicone residues on the polyamide surface. The Reference sample contains 95.6 ± 1.4% silicone, suggesting the preferential interaction of the silicone strands with the polyamide surface. The PSA attach process resulted in a surface silicone concentration of 97.2 ± 1.7%, which is statistically comparable with the reference sample, indicating the minimal effect of the PSA. Thermal treatment at 125 °C for 7 h resulted in a surface concentration of 60.7 ± 2.4%, indicating the thermally-induced intercalation of the silicone strands into the PSA layer. These results suggest that the silicone strands have preferential interaction with polyacrylate at elevated temperatures. This interaction is possibly due to the disentanglement of the polyacrylate strands in the PSA layer, allowing for the mobility of the silicone strands into the adhesive body. Interestingly, after the thermal treatment and the drop test, the surface silicone content increased to 91.5 ± 2.3%. This treatment occurs at room temperature, further supporting the mechanistic insight of preferential adhesion with the polyamide surface at room temperature via silicone de-intercalation. Short thermal treatment at 250 °C during reflow resulted in the surface concentration to decrease at 87.4 ± 1.1%. This is consistent with the thermally-induced silicone intercalation mechanism into the adhesive layer at elevated temperatures. The degree of silicone intercalation is dependent on the duration of the thermal treatment.

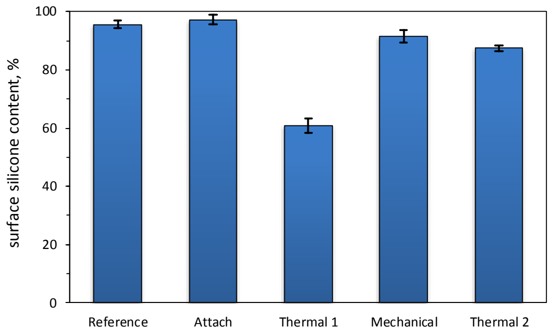

Fig. 4. Silicone content of the polyamide surface after each process step.

IV. CONCLUSION

Surface determinants identified through TOF-SIMS analyses reveal the variable concentration of the silicone in the adhesion interface of the polyamide-polyacrylate system. The residual silicone has preferential interaction with the polyamide surface at room temperature, but due to the thermally-induced mobility of the polyacrylate and silicone strands at higher temperature, the preference shifts to silicone intercalation into the polyacrylate layer. The intercalation is reversible.

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