Silicone release liners, problem solving in release applications

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Introduction

Elkem Silicones is one of the foremost fully integrated silicone manufacturer. Elkem Silicones offers a comprehensive range of silicone products in the sectors of release coating, textile, engineering elastomers, healthcare, specialty fluids.

The Silcolease® range is unique in its ability to cover all technologies used in silicone release coatings: Solventless thermal, Solventless radiation, Emulsion and Solvent.

The problems that are commonly associated with end use of Silcolease® products for release applications against pressure sensitive adhesives are relatively few in number, but each has many contributing factors, and many problems are interlinked. This document tries to relate problems to possible causes or what should be analyzed and gives advice on how to resolve certain problems.

Problems commonly encountered

1. Release Force
2. Bad curing/Migration
3. Change of subsequent adhesion
4. Printability
5. Rub-Off
6. Slip/Friction
7. Die-cutting
8. Bath life/gelling
9. Gloss Problems
10. Oven dust
11. Blocking
12. Telescoping
13. Laminate curl
14. Emulsion coating problems
Release Force

When you have a release force problem, a question to ask yourself is “Have I got the right combination of raw materials?”. You should always keep in mind that not all silicones and adhesives give the same release and that the chemical and physical nature of your liner substrate can have a significant impact on the release force.

Here is an example of how the release force can change with silicone polymer type and the speed of peel for any given adhesive system. Polymer A giving similar release force at any speed whilst Polymer C rapidly increases with speed of release.

<table>
<thead>
<tr>
<th>Stock Face</th>
<th>Adhesive</th>
<th>Silicone</th>
<th>Paper (or filmic substrate)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Chemical nature</td>
<td>Polymer type &amp; stoichiometry</td>
<td>Smoothness</td>
</tr>
<tr>
<td></td>
<td>Coat weight</td>
<td>Coat weight and uniformity</td>
<td>Porosity</td>
</tr>
<tr>
<td></td>
<td>Residuals (solvents &amp; component migration)</td>
<td>Surface energy &amp; release additives</td>
<td>Stiffness</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>pH &amp; residual chemicals</td>
</tr>
</tbody>
</table>

### Figure 1. What is release coatings?

### Figure 2. Release Force vs. Peeling Speed
### Tight Release

The most fundamental property of the release coating is the requirement of giving suitable release of the adhesive material that it is protecting. The most common of problems is that release is not easy enough and this fact can be caused by any number of the conditions that follow. When we talk about tight release this can be at:
- High speed only
- Low speed only
- All speeds

The following factors have been found to affect the release, and some understanding of these factors will be essential for each case to be investigated:

<table>
<thead>
<tr>
<th>Silicone Release Layer</th>
<th>Adhesive</th>
<th>Substrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Polymer type</td>
<td>- Chemical nature</td>
<td>- Type of paper or film</td>
</tr>
<tr>
<td>- Polymer properties</td>
<td>- Modulus</td>
<td>- Porosity</td>
</tr>
<tr>
<td>- Stoichiometry</td>
<td>- Surface energy</td>
<td>- Smoothness</td>
</tr>
<tr>
<td>- Modulus</td>
<td>- Coating weight</td>
<td>- Stiffness / Flexibility</td>
</tr>
<tr>
<td>- Coating weight</td>
<td>- Uniformity of coating weight</td>
<td>- Calliper consistency</td>
</tr>
<tr>
<td></td>
<td>- Smoothness of coating</td>
<td>- Surface energy</td>
</tr>
<tr>
<td></td>
<td>- Crosslink density</td>
<td>- pH</td>
</tr>
<tr>
<td></td>
<td>- Film continuity</td>
<td>- Internal bond strength</td>
</tr>
<tr>
<td></td>
<td>- Cure mechanism</td>
<td>- Surface absorbency</td>
</tr>
</tbody>
</table>

- Surface energy
-Extent of cure
-Level of release additive

- Residual solvent level
- Component migration

- Surface absorbency
- Residual chemicals from manufacturing
However, many of these factors will be well known when any problem is reported. From the silicone side of the problem it is essential to establish for any complaint of tight release that the following factors are known:
- Coat weight
- Coverage (pinholes etc....)
- Cure
- Silicone formulation / composition
- Release specificity to particular adhesives

By establishing these basics, it should then be possible to decide on which area to concentrate to explain the occurrence of tight release. Only from an understanding of these factors can a solution be proposed with any confidence.
Release instability/Variation in release values

Release values are measured usually after a certain set period of time for either silicone coated papers or for laminates. The variation in the release test results (typically FINAT TM 3, 4 & 10) can vary by at least 10% simply due to experimental error/variation and the vast number of factors affecting a single measurement. Obviously each construction will have different parameters with varying degrees of effect, and so there will be cases of perfect reproducibility and others of huge variation. These variations are unavoidable, in practice, when measuring a liner release against the most relevant adhesive.

The release values vary to differing degrees, but on certain occasions an increase or reduction of release values over time (for either the liner alone or the laminate) can cause problems for end applications. In summary, the main effects are as follows:

- **Lock-up** - Release values increase with age of laminate
  - Because of an interaction between “acrylic” adhesives and residual silicone reactive groups, by poor coverage of silicone, poor silicone cure, poor adhesive drying, silicone or adhesive formulation (migration of tackifier or resins in adhesive or silicone).

- **Drop-off** - Release values decrease with age of laminate or paper
  - Poor cure of silicone (migration)
  - Hydrolysis of excess crosslinker (SiH) groups
  - Migration from face or backing substrate (particularly PVC)
  - General adhesive degradation

- **Reel to reel** - Where inconsistent values are seen from one run to another
Too Low Release

- Silicone formulation (e.g. not enough tight RCA for solventless silicone)
- Coat weight too high
- Under-cure (migration)
- Adhesive problem (drying, formulation, de-wetting...)

Two other common release phenomena can occur, namely Zippy release, otherwise known as a Slip-Stick type of release, and Peak release, an initial high release force.

Zippy Release

This problem manifests itself mainly in low speed release. The average release can be good but the release curve is very spiky with a large variation between minimum and maximum values for a peel test. The reasons for the zippy release are most commonly very similar to those causing peak release problems but when compared to a smooth release profile sample, a slow speed zippy release will also show more “zippy” nature at high speed.

Most common causes are:
- Adhesive modulus, tackifier level and type, etc.
- Pinholes / poor coverage of silicone
- Chemical or physical interactions
- Backing and face substrates stiffness and strength
- Silicone modulus

Peak Release

Usually appears as a very tight release peak at low to medium peeling speed.

Most common causes are:
- Adhesive modulus
- Pinholes/poor coverage of silicone
- Chemical or physical interactions
- Backing and face substrates stiffness and strength
- Silicone modulus
- Die-cutting problems
Bad Curing/Migration

Cure is difficult to define as an absolute factor as the only true effect is whether the laminate performs adequately under the end use. Some laminate constructions are capable of accepting a degree of under-cure that in other more sensitive areas would cause total rejection of the laminate.

- Bad cure (under-cure/smear/migration/extractables) can be most commonly attributed to:
  - Machine speed too fast
  - Temperature too low (oven air velocities, etc.)
  - Coat weight
  - Silicone formulation (SiH/SiVi, catalyst level, etc.)
  - Substrate - catalyst poisoning
  - Age of bath
  - Other potential inhibitors of reaction (from machine, raw materials, etc)
  - Solvent type & grade - volatility, inhibition (Sulphur free grades)

- Silicone extractables as a measure of cure

A commonly used industry standard but it is important to understand the relevance of this test!
  - Non-chemically linked materials are extracted from the cured network by an organic solvent and measured analytically
  - Some silicone materials can be non-chemically linked but form a stable and reliable release performance
  - Extractable species are not necessarily the same thing as migrating species
  - Many applications can be unaffected by, or even benefit, from a small amount of migrating silicone:
    - Bitumen
    - High adhesive coat weight applications
    - Zippy release
Change of subsequent adhesion

Sometimes the tack and adhesive performance can be changed after contact with the silicone coated substrate. The single most important factor is likely to be silicone migration caused by poor curing, but this is not the only effect. The most important factors are:

- Low subsequent adhesion
  - Under cure of silicone leading to migration
  - Other migratory material from backing or top face or from within adhesive itself
  - Very rough silicone surface can reduce the smoothness of the adhesive
  - Adhesive deterioration with age, atmospheric exposure, moisture content of paper etc...
- Higher subsequent adhesion
  - Very smooth silicone surface can improve the smoothness of the adhesive
  - There can be adhesive changes due to contact with silicone such as tackifier migration etc...

Printability Problems

These can be either on the back of the silicone paper, or on the top face of the laminate.
- Off-line: Silicone under-cure causing migration to the back of the paper (and then potentially on to top face).
- In-line: Silicone volatiles or misting.
- Substrate problems: penetration of silicone through paper.
- Silicone or other sprays used around printing machines.
- General contamination from a dirty machine, or another source.
Rub-Off

This can be seen immediately or on ageing of the silicone coated substrate:

- Silicone formulation (can be anchorage or adhesive failure, or cohesive failure of the cured silicone):
  - Too low crosslinker level
  - Wrong crosslinker type
  - Poor cure
  - Crosslink density of the silicone (more relevant for solventless silicone grades)
- Substrate (will lead to anchorage or adhesive failure):
  - Too smooth or too low in porosity
  - Surface energy (films)
  - Surface inhibition
  - Lack of adequate corona or other surface treatment
- Type of solvent used for dilution
- Aged rub-off can be due to:
  - Post curing
  - Re-orientation of silicone matrix
  - Migration to substrate surface of disruptive materials
  - Surface energy changes

Slip/Friction

The lack of friction or slipperiness of the silicone surface can cause production problems further down the converting line.
These can be controlled to some extent by:
- Silicone type: solvent Vs solventless, hardness, etc.
- Silicone cure: migrating silicone
- Coat weight
- Additives
Die Cutting

Not usually a silicone problem but matrix removal may highlight release problems. Soft silicones can be more easily damaged by die-cutting process and could lead to subsequent problems.

Bath Life/Gelling

Bath life problems can lead to bad release, bad cure, rub-off or general runnability problems caused by viscosity changes and gelling. The main reasons for bath life problems are:

- Silicone formulation (too reactive - high catalyst, high crosslinker or low inhibitor levels)
- Solvent type and grade (volatility, inhibiting nature)
- Ambient temperature
- Inhibitor evaporation or reaction with other material
- In emulsions - droplet coalescence leading to micro-gelling
- Temperature on the machine generated by coating head or bath handling apparatus
- Bath size and storage

Gloss Problems

Gloss levels are often used as an indicator of quality. Loss of gloss through a coating run could suggest bath life problems or a general change in process conditions. Gloss can also have some useful properties in terms of release level and subsequent gloss level of the adhesive leading to change in immediate tack. Low gloss on a silicone coated substrate can be due to:

- Paper surface (too rough or too porous)
- Silicone cure often associated with aged baths
- Too much heat on PE Kraft can cause PE to fuse (as can wrong grade of PE)
- Micro-gels in silicone emulsions
- Adhesive / silicone interaction causing surface damage
- Rub-off
Oven Dust

Dust can come from many sources such as paper. When we consider silicone dust, this is usually only residue from tin catalyst in the older style polycondensation systems, or volatile silicones which have been thermally degraded to silica. This degradation normally only occurs when direct flame burners are used to heat the drying / curing air which is then recirculated through the ovens several times. Dust can also be a problem on paper machines using direct contact drying cylinders.

Blocking

This problem was a severe problem with older tin catalysed systems. It is mainly seen with two side silicone coated substrates with today’s more commonly used polyaddition silicones. There is a strong bond between the two sides of the paper which leads to the surfaces sticking together under pressure in the reel. This is sometimes only a nuisance in terms of noise (when re-reeling / slitting / converting), but in its worst case can cause damage to the silicone surface or even cause paper tears.

- Blocking is most commonly caused by:
  - Under-cure of silicone followed by interaction between surfaces
  - Softness of silicone coating
  - Static problems
- Blocking can be made worse by:
  - Very tightly wound reels
  - Certain re-humidification conditions causing swelling of fibres after reeling
  - Plasticising materials in the silicone formulation

Telescoping

The telescoping of the reel edges can be caused by several factors:

- Silicone formulation:
  - Lack of slip properties
  - Too low coefficient of friction
  - Variation in thermic properties at curing
- Coating conditions:
  - Inappropriate coatweight
  - Nature of the network (too hard or too soft)
- Problem with the machine:
  - Misalignment of incoming webs or winder rolls
  - Unsymmetrical diameter of paper roll
Laminate Curl

Excessive drying of paper based substrates can lead to curl in the laminate. To avoid this effect either a low temperature thermal cure silicone, good rehumidification or a less sensitive substrate must be used. This curl problem is even more significant with emulsion based silicones.

Emulsion Coating Problems

There are several different problems occurring due to the use of emulsion silicones in particular:
- Wrinkles/cockling effect of water on paper
- Curl due to moisture imbalances caused by excessive drying of papers
- Wetting problems due to surface tension or viscosity of emulsion coating bath
- Higher energy to dry water can give curing problems
- Foaming, micro-gelling in droplets, basic emulsion stability and effects such as coalescence are fairly unique to emulsions
- Solids increase due to preferential absorption of water into paper

Summary

The problems listed in this document are those most commonly facing the silicone release liner side of the self-adhesive laminate market. The testing of each variable suggested can be done according to standard test methods either from industry standards (such as FINAT, TAPPI, and TLMI) or from other set methods. Elkem Silicones has available a full list of test methods that are followed to test all parameters discussed. Other test regimes are used, such as standard FINAT methods (for all laminate related tests), or generally accepted tests used within the industry.
Elkem Silicones

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