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Developing a Test Method to Determine the Cause of a Cracking Cosmetic Product

Background

Sometimes a lab customer's troubleshooting needs are easily met with standard analytical tools and technology; sometimes they involve questions only answered by developing new tests and methods. The latter was the case when Teel Plastics was approached by a customer about an AES cosmetic pen barrel with a polypropylene end cap that was failing under standard quality control heat tests. Since Teel manufactured the barrel, Teel agreed to help troubleshoot the problem, keeping with our practice of making our analytical lab, Teel Analytical Laboratory (TAL), available to all manufacturing customers for analysis. The project proved to be an opportunity for the Teel's lab to develop custom methods of investigation. In the end, Teel was able to provide answers for our customer about the causes of the cracking, as well as actionable information suggesting ways to prevent the cracking in the future.

The Problem

To assess the longevity of their products, cosmetics companies typically conduct accelerated shelf-life tests, also referred to as temperature conditioning. This quality control measure involves weighing a cosmetic container filled with cosmetic, heating it in an oven, and weighing it periodically over the course of months to evaluate the level of cometic lost to evaporation over time. In this case, the customer had filled their pen barrels with cosmetic, capped them, and placed them into an oven at 50°C. After a week, the customer noticed cracks forming on the barrels at the seam between the cap and barrel, something that would never typically happen.

Initial Hypotheses

TAL's initial thought was that the problem must be one of faulty dimensions. They believed that either the cap was too large or its inner diameter was too small. That is, as the pen and cap expanded under heat, the cap must have pressing onto the barrel too strongly either because:

1. The cap was too thick, the excess material leading to excessive expansion

or

2. The cap's inner diameter was designed to fit too snugly barrel initially, leading to easy breakage as it expanded.

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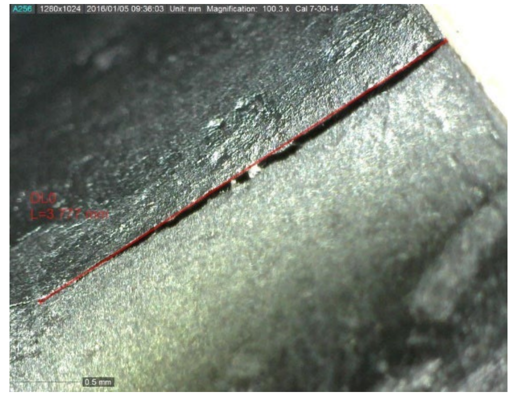
Testing



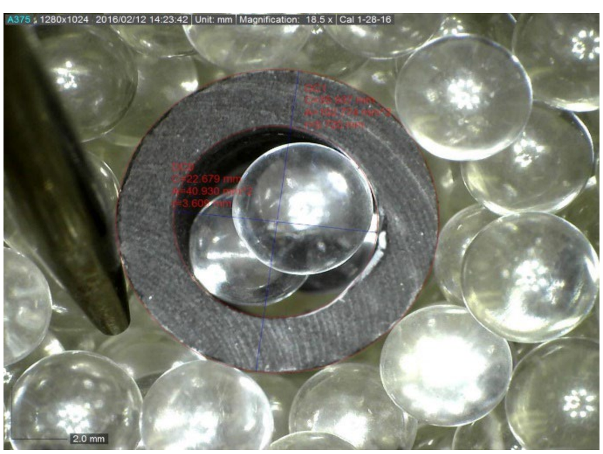
Cracking present on heat-tested AES barrel sample.

TAL started the testing process by first replicating the customer's observations, mimicking the same heat conditioning test they had conducted. A sample pen filled with cosmetic was capped and placed in an oven at 50°C and monitored every 24 hours, while an identical control sample was left out of the oven and monitored on the same schedule. At the first 24-hour check of the heated sample, liquid was leaking out of the barrel between the polypropylene end cap and barrel seam. This liquid was analyzed by FT-IR testing and found to have similarities to silane, a solvent. After a week, TAL observed cracking in the heated sample at the seam where the initial leaking was observed, just as the customer had said.

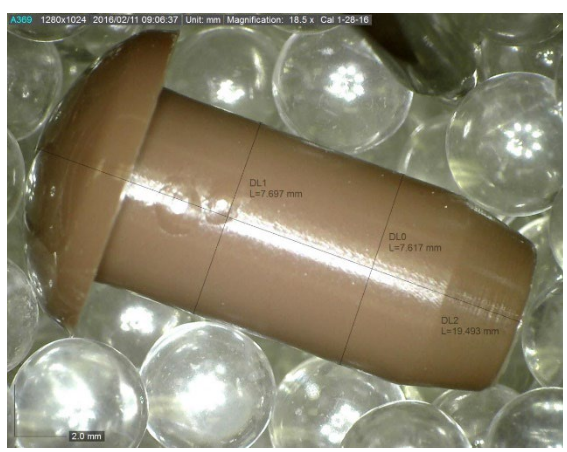
TAL concluded that the cracks may have been the product of environmental stress cracking, which occurs when there is a combination of applied pressure and another contributing environmental factor. In this case, TAL believed the silane, solvent-based cosmetic was likely the environmental factor weakening the AES barrel material enough to lead to cracking as the cap and barrel expanded under heat.

To verify, TAL wanted to examine what happened to the barrel and cap as they expanded when no cosmetic was present. This would help confirm whether the solvent was necessary for the cracking to occur or if the cracking was the result of heat expansion alone. Further, the fact that the cracking only occurred at the seam where the cap and barrel met was a point of interest, making TAL curious if there might be some way the polypropylene cap and AES barrel interacted under heat that contributed to the cracking.

To investigate, TAL conducted another test of their own design. First, they placed a polypropylene cap and an AES barrel without cosmetic in two separate containers filled with glass beads. They then used a hot plate to gradually heat up the parts within each container, the glass beads conducting heat to the samples. TAL then observed with a microscope how the cap and barrel behaved as they were heated, taking measurements at 30°C, 40°C, and 50°C.



AES barrel sample with dimensions during glass bead heat test.



Polypropylene cap sample with dimensions during glass bead heat test.

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Over the course of the test, the polypropylene cap expanded more than the AES barrel, leading TAL to conclude that another possible reason for the cracking was merely the pressure of the polypropylene cap expanding against the AES barrel. The AES barrel, being a foamed product, would be likely to fail sooner than the polypropylene cap.

AES Barrel Dimensions over the Course of the Glass Bead Heat Test

Ambient (mm)		30° C (mm)		40° C (mm)		50° C (mm)		Radius Increase (mm)	
ID	OD	ID	OD	ID	OD	ID	OD	ID	OD
7.218	11.440	7.238	11.460	7.260	11.454	7.266	11.480	0.048	0.040

Polypropylene Cap Dimensions over the Course of the Glass Bead Heat Test

Ambient (mm)		30° C (mm)		40° C (mm)		50° C (mm)		Width Increase (mm)	
Width 1	Width 2	Width 1	Width 2	Width 1	Width 2	Width 1	Width 2	Width 1	Width 2
7.617	7.697	7.775	7.851	7.786	7.821	7.791	7.873	0.174	0.176

As their theory that the solvent played a necessary role came into question, more investigation would be needed. As a final test, TAL conducted the same heat conditioning test as at first, but this time with a sample not filled with cosmetic. The team heated the sample to 50°C and monitored it every 24 hours for one week. In the end, no cracking was observed on the unfilled sample. It was clear the presence of the solvent-based cosmetic was in fact necessary to produce the cracking.

Conclusion

TAL concluded that the cracking observed in the parts was environmental stress cracking. The stress-induced cracking from heat expansion was accelerated by a solvent in the cosmetic formulation.

With this information, TAL helped move the customer toward a solution. Solutions for the customer would likely be one of four options:

1. Reduce the heat of the initial conditioning test. Perhaps 50°C was too aggressive and the product would perform without issue at a slightly lower temperature, and perhaps that would be all that was needed to assess the quality and reasonable life of the product.
2. Reduce the dimensions of the barrel or cap. Perhaps the dimensions were at a tipping point and a slight size reduction would solve the problem.
3. Use a cosmetic with a different solvent base. Perhaps another solvent wouldn't act as aggressively on the AES barrel as the current one.
4. Use different materials for the cap and barrel that would work better together. Perhaps the strength and/or heat expansion properties of other materials would prevent the cracking.

The project gave TAL the opportunity to demonstrate its versatility and its thoroughness in answering troubleshooting inquiries. TAL took the time and effort to meet the customer's needs and provide actionable information, even developing custom testing procedures in the process.

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