Nanomechanical Testing of Thermal Transfer Print

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INTRO

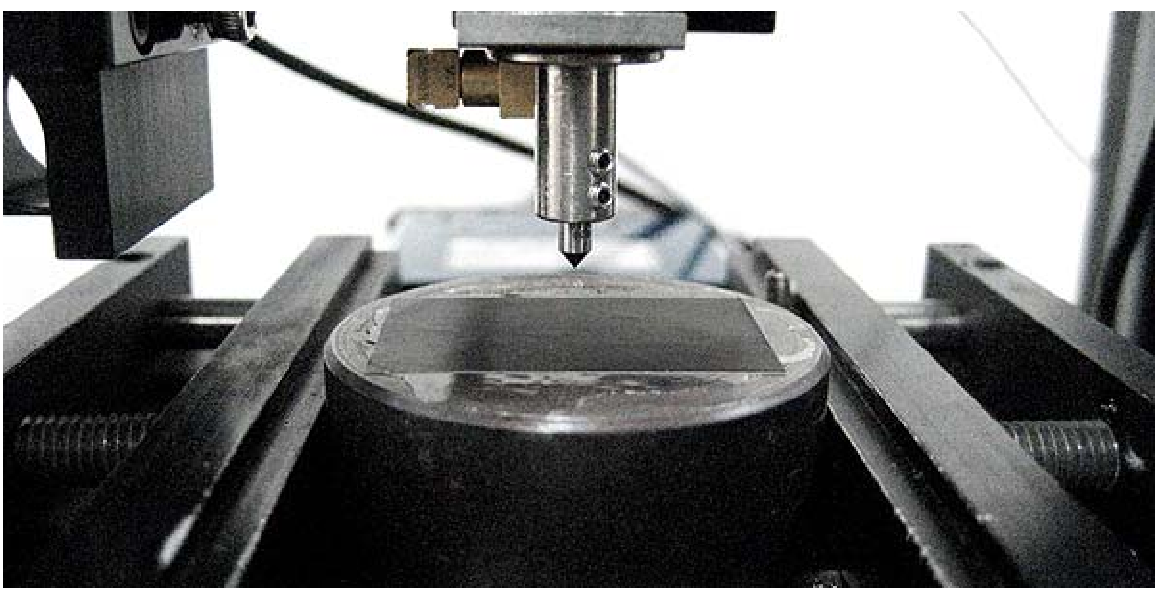
Digital thermal transfer printing, unlike direct thermal printing, uses the process of melting a coated ribbon to the material on which the print is applied. There are 3 main types of thermal ribbons: wax, wax/resin or pure resin and are almost always black. These different types have varying qualities to survive harsh environments and are selected to print on different material surfaces.

IMPORTANCE OF NANO SCRATCH TESTING FOR QUALITY CONTROL

Digital thermal transfer printing is used primarily for its print durability in applications such as bar codes and printing labels. These applications demand high resistance in harsh environments for long periods of time. Therefore, the formulas related to different types of digital thermal transfer ribbons should have a known/tested resistance to scratch and adhesion failure. By using the nano scratch testing method the failure of digital thermal transfer printing can be tested and compared to identify the most durable formulation.

MEASUREMENT OBJECTIVE

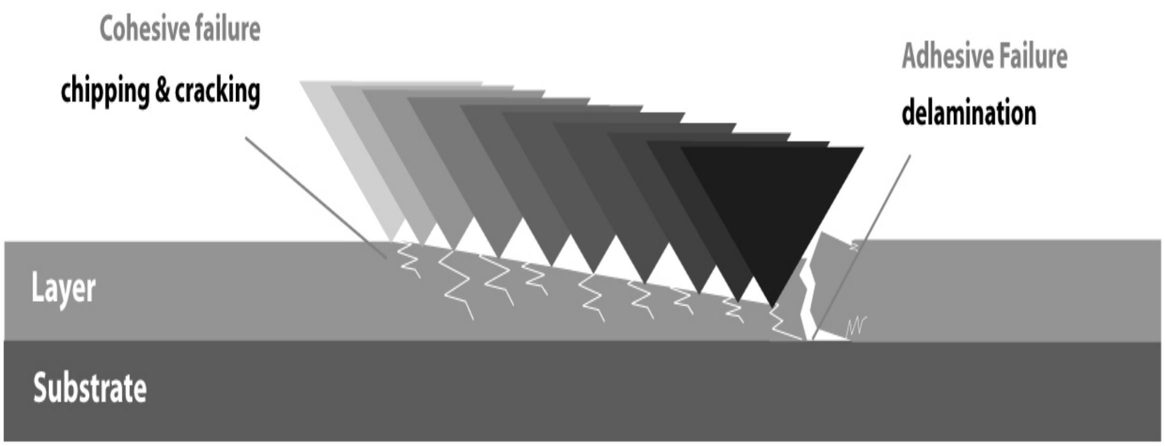
We must simulate the process of scratching in a controlled and monitored manner to observe sample behavior effects. In this application, the Nanovea Mechanical Tester, in nano scratch testing mode, is used to measure the load required to cause failure to 3 micron wax/resin prints on coated paper. A 2μm 90° cone diamond tip stylus is used at a progressive load ranging from 0.10 mN to 0.20 mN to scratch the printed surface. Points of failure will be reviewed. In addition, we have also used nanoindentation mode to obtain hardness and elastic modulus of the sample prints.



MEASUREMENT PRINCIPLE:

The scratch testing method is a very reproducible quantitative technique in which critical loads at which failures appear are used to compare the cohesive or adhesive properties of coatings or bulk materials. During the test, scratches are made on the sample with a sphero-conical stylus (tip radius ranging from 1 to 20μm) which is drawn at a constant speed across the sample, under a constant load, or, more commonly, a progressive load with a fixed loading rate. Sphero-conical stylus is available with different radii (which describes the "sharpness" of the stylus). Common radii are from 20 to 200μm for micro/macro scratch tests, and 1 to 20μm for nano scratch tests.

When performing a progressive load test, the critical load is defined as the smallest load at which a recognizable failure occurs. In the case of a constant load test, the critical load corresponds to the load at which a regular occurrence of such failure along the track is observed. In the case of bulk materials, the critical loads observed are cohesive failures, such as cracking, or plastic deformation or the material. In the case of coated samples, the lower load regime results in conformal or tensile cracking of the coating which still remains fully adherent (which usually defines the first critical load). In the higher load regime, further damage usually comes from coating detachment from the substrate by spalling, buckling or chipping.



The scratch test gives very reproducible quantitative data that can be used to compare the behavior of various coatings and substrate materials. The critical loads depend on the mechanical strength (adhesion, cohesion) of a coating-substrate composite but also on several other parameters: some of them are directly related to the test itself, while others are related to the coating-substrate system.

The test specific parameters include:	The sample specific parameters include:
• Loading rate • Scratching speed • Indenter tip radius • Indenter material	• Friction coefficient between surface and indenter • Internal stresses in the material For bulk materials • Material hardness and roughness For coating-substrate systems • Substrate hardness and roughness • Coating hardness and roughness • Coating thickness

Means for critical load determination

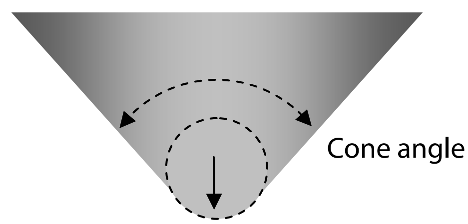
Microscopic observation
This is the most reliable method to detect surface damage. This technique is able to differentiate between cohesive failure within the coating and adhesive failure at the interface of the coating-substrate system.

Tangential (frictional) force recording
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses to failures are very specific to the coating-substrate system in study.

Depth Sensing
Sudden change in the depth data can indicate delimitation. Depth information pre and post scratch can also give information on plastic versus elastic deformation during the test. 3D Non-Contact imaging such as white light axial chromatism technique and AFM's can be useful to measure exact depth of scratch after the test.

Test Parameters

Sample	Sample 1 and 3	Sample 2
Load type	Progressive	Progressive
Initial Load	0.10mN	0.10mN
Final Load	20 mN	15 mN
Loading rate	40 mN/min	30 mN/min
Scratch Length	2 mm	2 mm
Scratching speed, dx/dt	4 mm/min	4 mm/min
Indenter geometry	90° cone	90° cone
Indenter material (tip)	Diamond	Diamond
Indenter tip radius	2 µm	2 µm



Sphero-Conical Indenter

Results

Summary table of main numerical results:

Sample	Initial Delamination [mN]	Complete Delamination [mN]
1	2.28 ± 0.85	11.09 ± 0.66
2	3.84 ± 0.42	11.44 ± 0.86
3	4.81 ± 0.12	15.44 ± 1.93

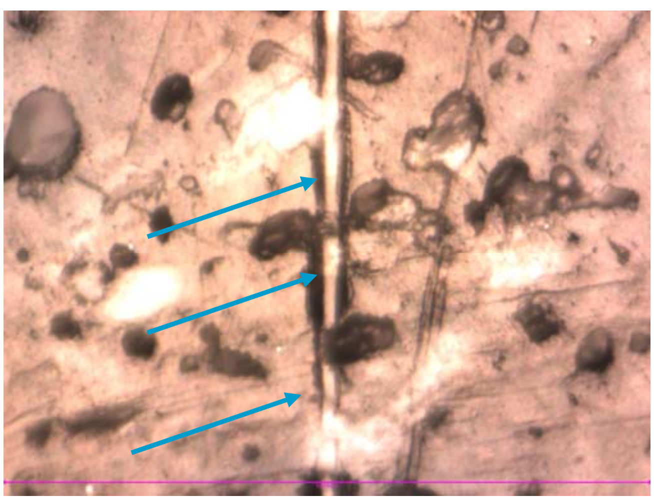
Results –Sample 1

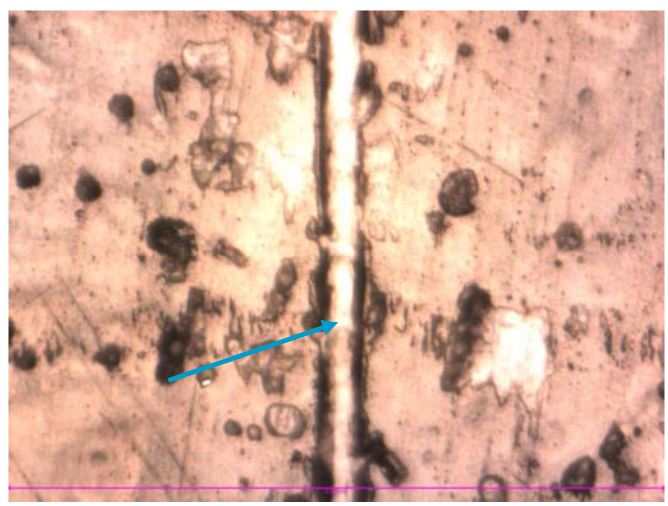
NOTE: Because the ink coating on Sample One was unevenly distributed, the scratch did not demonstrate a clear delamination

Scratch	Initial Delamination [mN]	Complete Delamination [mN]
1	1.59	10.32
2	2.02	11.52
3	3.22	11.42
Average	**2.28**	**11.09**
Standard Deviation	**0.85**	**0.66**

Chart of micrographs and critical failures – Sample 1

Initial Delamination: This is the point at which the coating is showing the first signs of failure. We begin to see this as it sporadically delaminates to the substrate





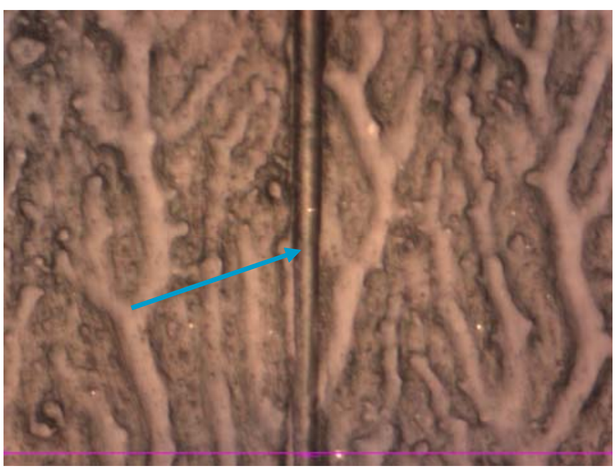
Micrographs of Initial & Complete Delamination
Sample 1 200x magnification (image width 0.249mm)

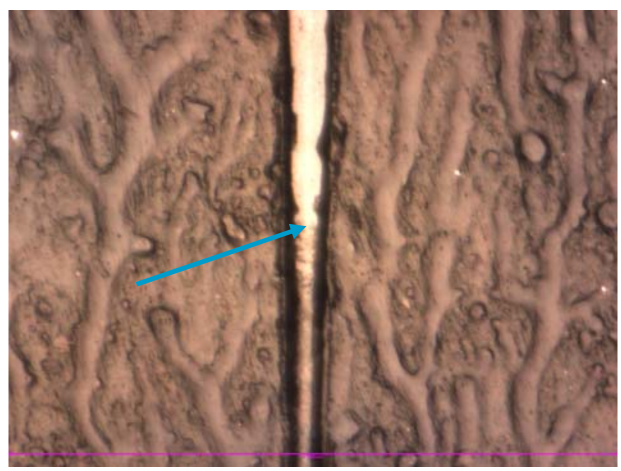
Results –Sample 2

Scratch	Sample 2	
	Initial Delamination [mN]	Complete Delamination [mN]
1	4.16	11.80
2	4.00	10.46
3	3.36	12.07
Average	**3.84**	**11.44**
Standard Deviation	**0.42**	**0.86**

Chart of micrographs and critical failures – Sample 2

Initial Delamination: This is the point at which the coating is showing the first signs of failure. We begin to see this as it sporadically delaminates to the substrate





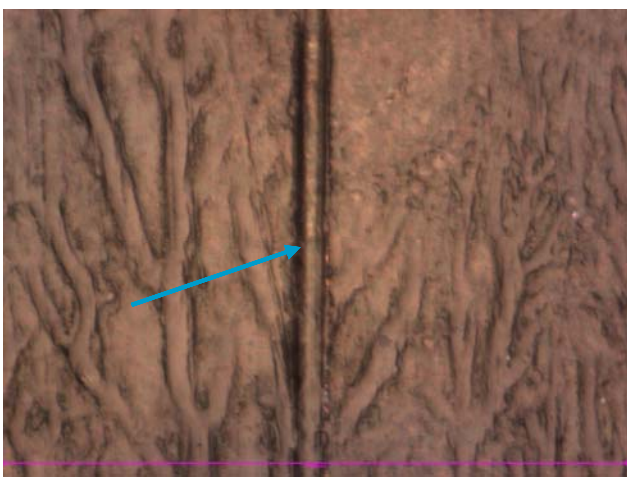
Micrographs of Initial & Complete Delamination
Sample 2 200x magnification (image width 0.249mm)

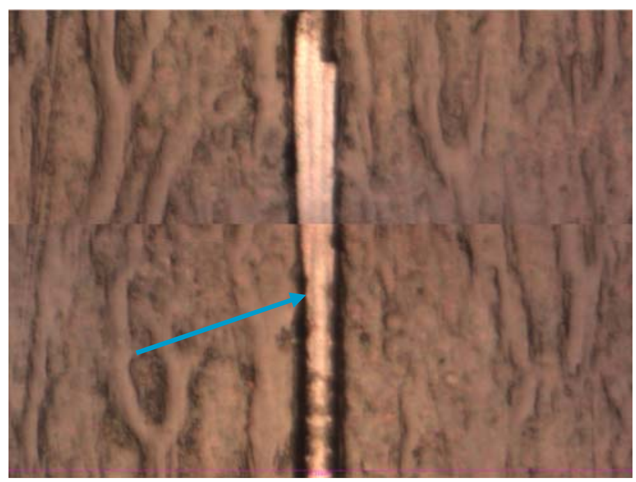
Results – Sample 3

Scratch	Sample 3	
	Initial Delamination [mN]	Complete Delamination [mN]
1	4.74	14.05
2	4.95	14.62
3	4.74	17.65
Average	**4.81**	**15.44**
Standard Deviation	**0.12**	**1.93**

Chart of micrographs and critical failures – Sample 3

Initial Delamination: This is the point at which the coating is showing the first signs of failure. We begin to see this as it sporadically delaminates to the substrate



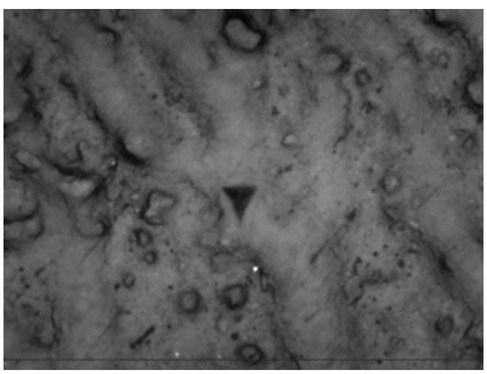


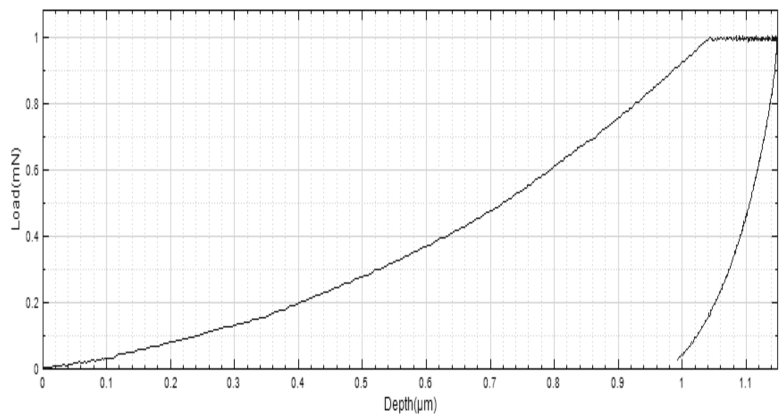
Micrographs of Initial & Complete Delamination
Sample 3 200x magnification (image width 0.249mm)

Additional Nanoindentation Results

Test Parameters	All Tests
Maximum force (mN)	1
Loading rate (mN/min)	2
Unloading rate (mN/min)	2
Creep (s)	20
Computation Method	ASTM E-2546 & Oliver & Pharr
Indenter type	BerkovichDiamond

Sample	Hardness [Vickers]	Hardness [GPa]	Young's Modulus [GPa]	Max Depth [nm]
1	4.40 ± 0.16	0.0465 ± 0.0017	1.35 ± 0.25	1037 ± 35
2	4.77 ± 0.62	0.0505 ± 0.0066	2.11 ± 0.37	970 ± 63
3	8.00 ± 0.74	0.0847 ±0.0078	3.06 ± 0.61	756 ± 27





Conclusion

As seen in our analysis, the scratch adhesion failure of the thermal prints has been identified. The technique identified the sample 3 as the most resistant and sample 2 as slightly better than sample 1. This data was then correlated with nanoindentation hardness measurements which also identified sample 3 as being the hardest. Other tests that could have been done include linear wear testing under various conditions to identify the effect of continuous rubbing on the print. This could have been performed with either the Mechanical Tester or with the Tribometer. Additionally, The 3D Non Contact Profilometer could have been used to image the surface of the print and or the volume loss of wear or scratch.

Learn More about the [Nanovea Mechanical Testers]