

SCRATCH ADHESION TESTING
OF BULK MATERIALS



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INTRODUCTION:

For most industries that manufacture products aimed at the general public, the cosmetic appearance of the product can be as important as its functionality. The overall appearance of the product will enhance the consumers' confidence in the product and will influence their opinion on its quality. Engineers working on these products must find a material or process that maximizes the resistance to scratching and marring, to keep the product looking intact for as long as possible.

To study the phenomenon of scratching, we must simulate the process of scratching in a controlled and monitored manner and observe the behavior of the samples under this scratch. In this case, Nanovea has worked with the company GrayGlass Global Solutions to study the resistance of their material to scratching and compare the results with their competitors' products. The samples tested are used for glass stovetop surfaces. In service, these parts will have metal pots and pans dragging on the surface with a high weight when full, which will make them subject to scratching and marring.

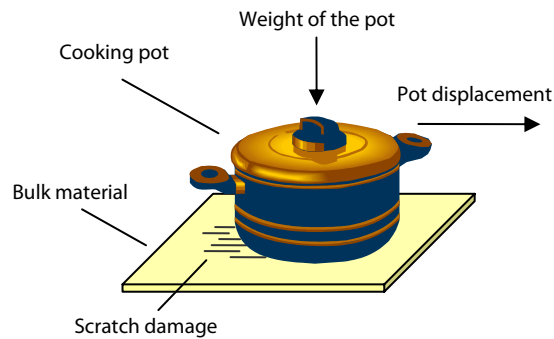


Figure 1 : Real life application

By using an instrumented scratch tester, we will be able to simulate the scratch process while taking out the randomness in the process (non uniform shape, actual local force, speed of displacement).

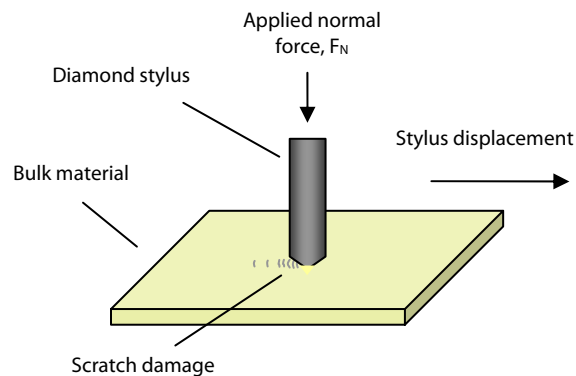


Figure 2 : Damage simulated by scratch testing

With this uniformed test, we can study the behavior of the samples under the scratch, and measure the critical loads at which observables failures occur, and use these values to quantitatively compare the scratch resistance of the samples.

Test Method:

The scratch testing method is a comparative test in which critical loads at which failures appear in the samples are used to evaluate the relative cohesive or adhesive properties of a coating or bulk material. During the test, scratches are made on the sample with a sphero-conical stylus (generally Rockwell C diamond, tip radius ranging from 20 to 200 μm) 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.

When performing a progressive load test, the critical load (L_c) is defined as the smallest load at which a recognizable failure occurs. The driving forces for damage in the scratch test are a combination of elastic-plastic indentation stresses, frictional stresses and the residual internal stresses. In the lower load regime, conformal or tensile cracking are typical failures visible on the surface. In the higher load regime, a sample with a coating can undergo a detachment of the coating from the substrate by spalling, buckling or chipping.

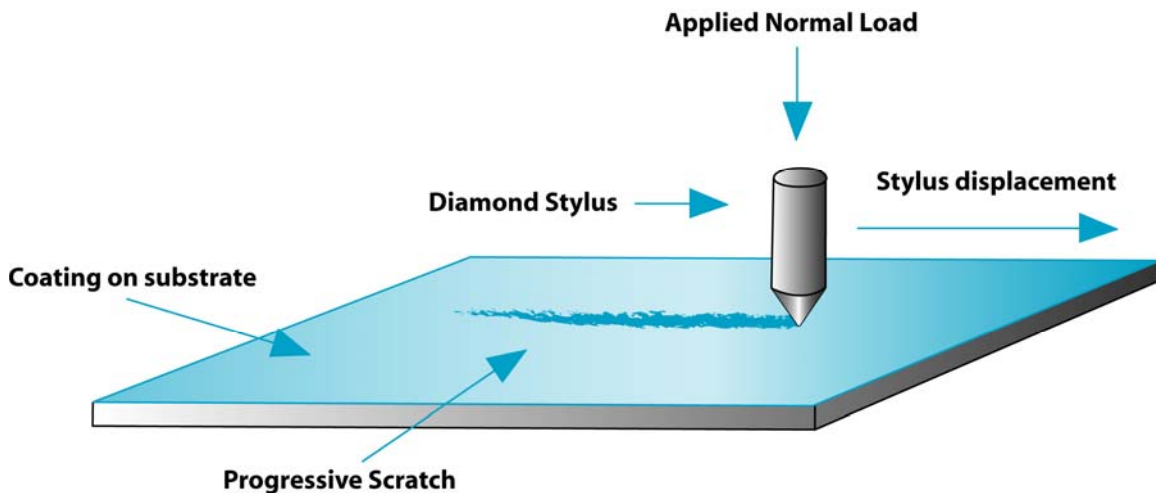


Figure 3 : Principle of scratch testing

Test Results:

In a first section, constant load scratches are showed to show the relation between microscopic damage and macroscopic damage, and in a second section, the data gathered during the test is presented.

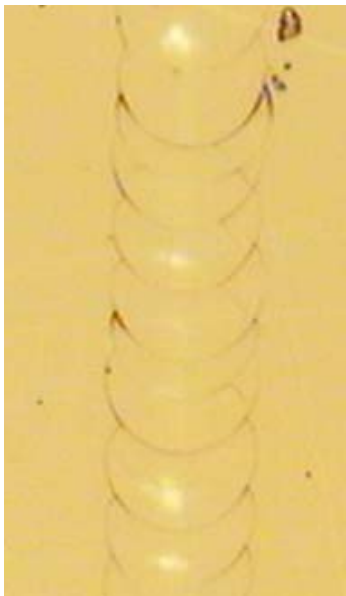
Constant Load Scratches

To relate the surface damage visible on the microscopic scale to the damage visible on the macroscopic scale, scratches at a constant load were performed. The following table shows the damage visible under the microscope at these different loads. For the progressive load scratch, the following test parameters are used:

Loading mode	Constant
Load	2, 3, 4 N
Scratch Speed	16.67 mm/min
Scratch Length	5 mm
Indenter Type	Sphero-Conical (120° cone angle)
Indenter Radius	50 μm

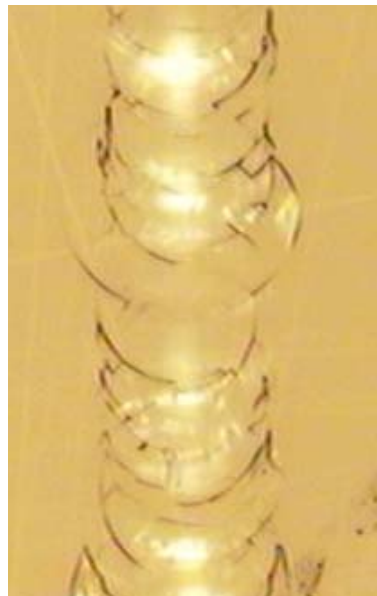
Constant Load Damage - magnification 200x (image width approx. 70 μm)

2N



The surface shows hertz tensile (circular cracks)

3N



The cracking is more severe, and the circular cracks are accompanied by radial cracks

4N



There is heavy damage as numerous cracks are superposed, and the scratch shows continuous whitening

The following photograph shows the macroscopic damage to the surface.

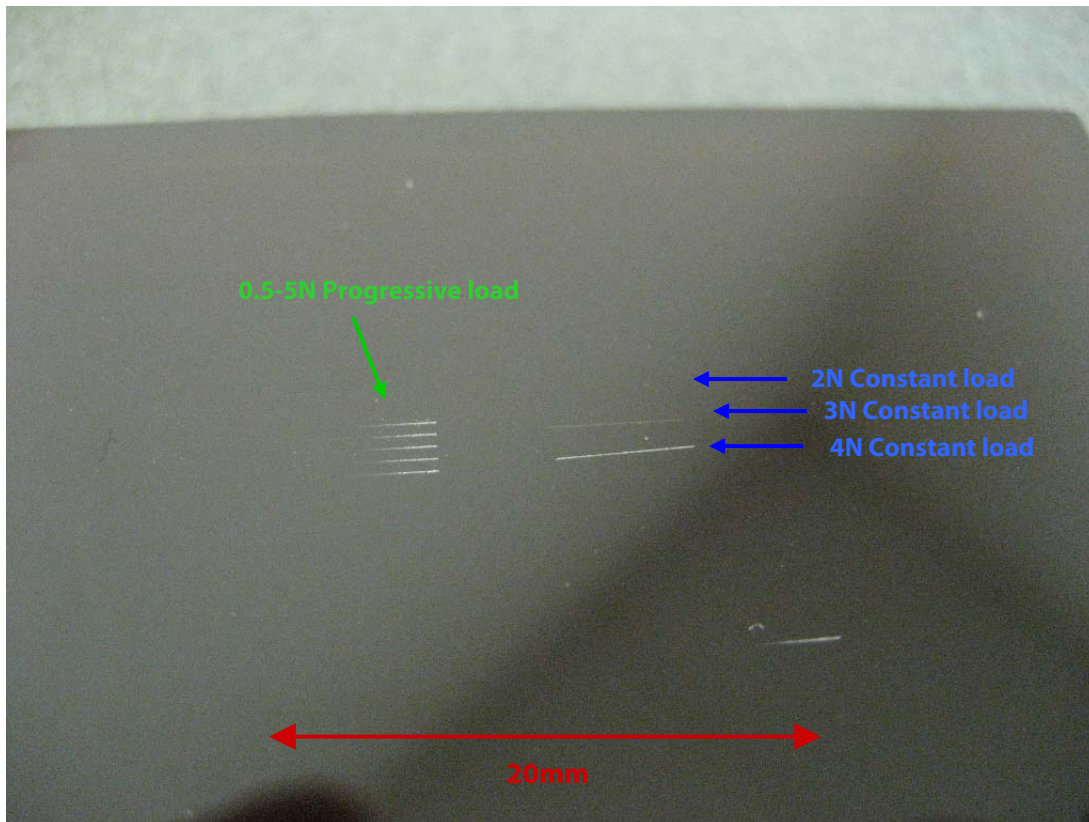


Figure 4 : Macroscopic damage of the scratches

Even though only the 3N and 4N scratches are visible on the photograph, the 2N scratch is visible when looking at the specular reflection on the surface. From this, we understand that the macroscopic scratches are in fact micro cracks in the material surface. Knowing this, we want to measure the load at which these failures occur when performing a progressive load scratch.

Progressive Load Scratches

To pinpoint the load at which the damage first occurs, in order to compare the samples' resistance to scratching, we perform a progressive load scratch which will be visually inspected. This section includes micrographs of different failure modes in the scratch, the critical loads at which the failures of interest occur, and a graph of coefficient of friction over scratch length. For the progressive load scratch, the following test parameters are used:

Loading mode	Progressive (linear)
Initial Load	0.5 N
Final Load	5 N
Loading Rate	15 N/min
Scratch Speed	16.67 mm/min
Scratch Length	5 mm
Indenter Type	Sphero-Conical (120° cone angle)
Indenter Radius	50 μ m

At loads under 1N, the indenter barely marks the surface, and causes no significant damage.



Figure 5 : Damage under 1N during progressive scratch

At a critical load ranging from 1 to 2N (depending on the sample), the first cracks occur on the sample surface.



Figure 6 : First circular crack during progressive scratch

At a critical load ranging from 2 to 3N (depending on the sample), the more severe cracking modes are visible.

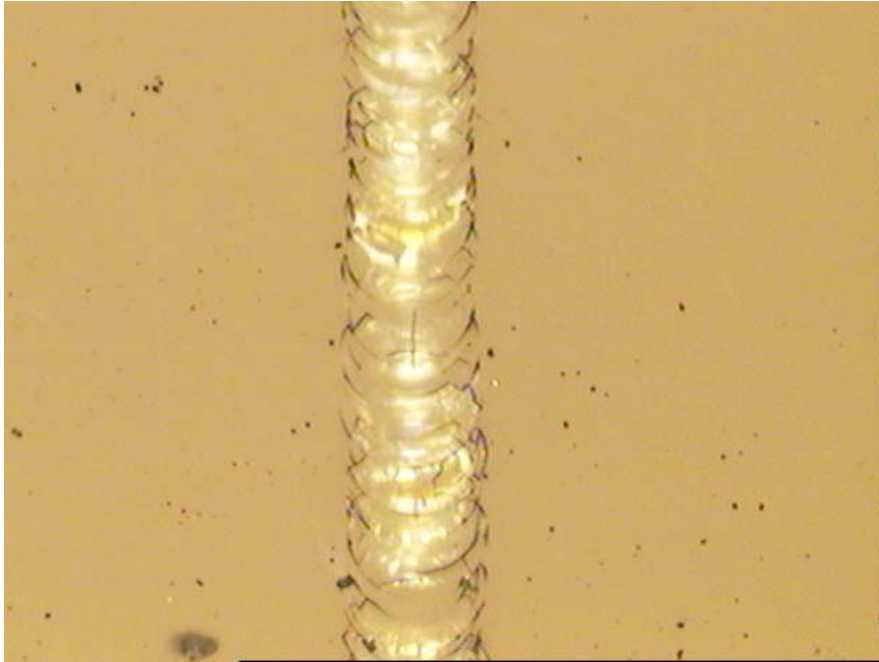


Figure 7 : More severe cracking with radial cracks visible during progressive scratch

At a critical load around 3N (depending on the sample), the heavy damage (superposed cracks) and continuous whitening is visible.

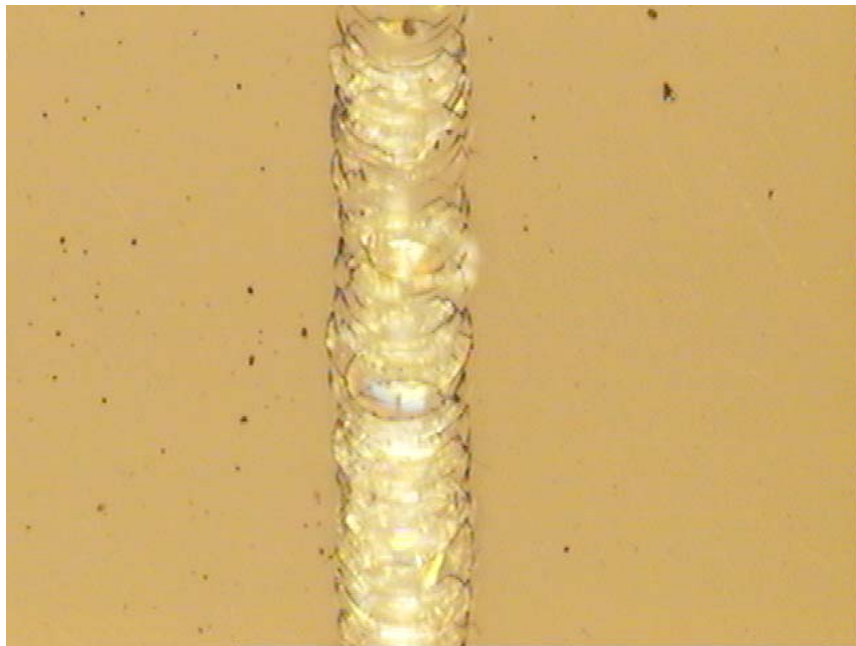
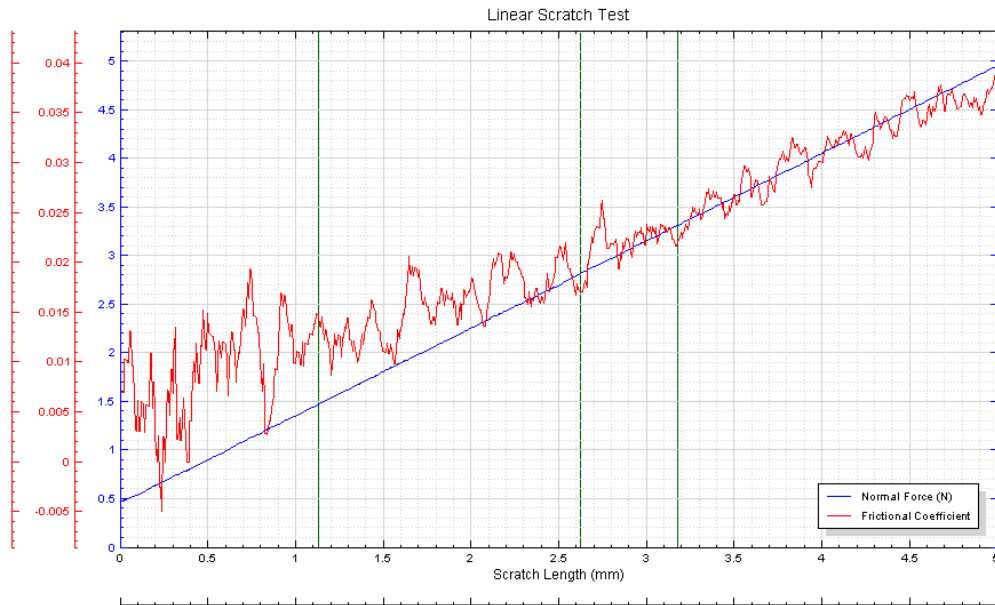


Figure 8 : Heavy damage and continuous whitening during progressive scratch

The following graph shows the recorded normal (vertical) and frictional (lateral) forces during the test as well as the coefficient of friction, over distance in mm. The vertical lines mark where the critical loads for both delaminations were recorded. On this graph, no significant steps or changes in slope are visible. In applications where material is removed from the sample by chipping, spallation or delamination, the coefficient of friction will change behavior at these points because of the work performed by the indenter on the sample. In this case, the friction graph was not used to determine the critical load. Only the visual inspection was used.



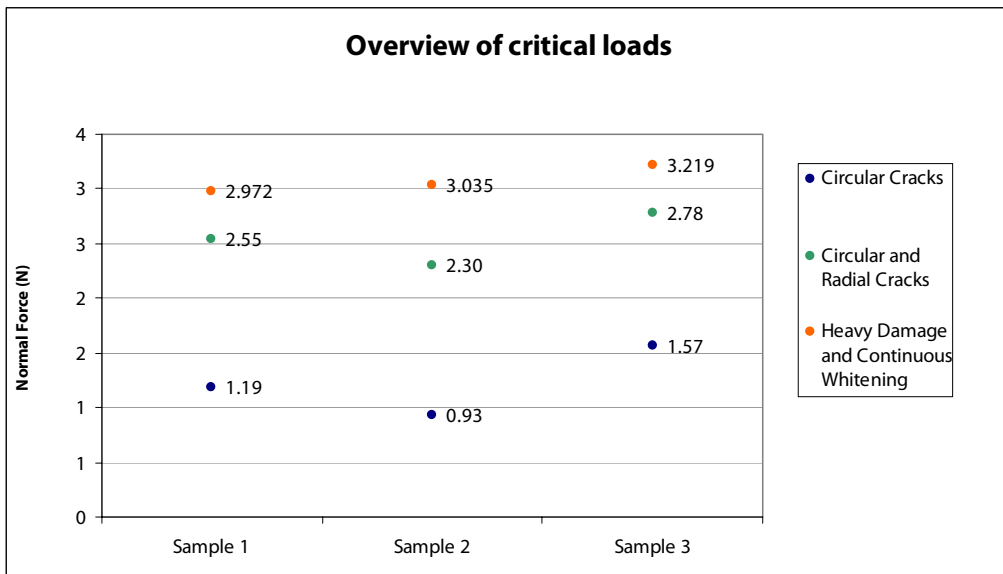
The following table shows the calculated results based off the critical loads identified in five scratches per sample.

Sample 1: Ceran®

Sample 2: KeraBlack®

Sample 3: Vitriam®

Sample	First Crack [N]		Forward Crack [N]		Severe Cracking and Continuous Whitening [N]	
	Value	Std Deviation	Value	Std Deviation	Value	Std Deviation
Sample 1	1.188	± 0.103	2.547	± 0.293	2.972	± 0.172
Sample 2	0.934	± 0.035	2.296	± 0.101	3.035	± 0.155
Sample 3	1.572	± 0.382	2.78	± 0.114	3.219	± 0.135



Discussion of the Results:

All three samples were tested five times with the same parameters. The critical loads are an indicator of the scratch resistance of these samples. If the critical load is higher, more force had to be applied to this sample for the same failure, meaning that the sample is more resistant.

Looking at the results, we can see that the critical loads of sample two are lower than those of sample 1, except for the heavy damage, which is slightly higher. Since the first two critical loads are clearer to identify visually, they are more repeatable and reliable than the third. This indicates a scratch resistance that is slightly higher for sample 1. When comparing sample 3 with the other two samples, we observe critical loads that are significantly, and consistently higher than those of the other two. This test clearly shows that sample 3 is more resistant to scratches and cosmetic defect.

Conclusion:

The Nanovea Micro Scratch Tester is able to mimic a real-life scratch and observe the micro cracks that make up that crack. By applying load in a controlled and closely monitored fashion, the instrument allows to identify at what loads the different failure modes in the scratch occur, in order to use these critical loads as a quantitative value for comparing scratch resistance between samples. This test was able to determine that the scratch resistance of this manufacturer's product was superior to those of its competitors. This test demonstrated was done on glass surfaces, showing typical failures of harder, more fragile surfaces. We can also test the scratch resistance of softer surfaces with the micro scratch tester. In some cases, no cracking will be visible, but the point of transition between elastic and plastic deformation, for example, might be visible.

Acknowledgement:

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