

Analysis of laboratory nitrile gloves: From pores to the surface

Relevant for: rubber, thin films, elastomers, atomic force microscopy, density testing, nanostructure analysis, surface roughness, surface area

The humble rubber glove is usually the first line of protection for any person working with toxic, hazardous and contagious materials or environments. This is especially true in the ongoing COVID-19 virus pandemic. While everyone agrees on their universal use, very little attention is paid to the type of material, surface topography or porosity of the glove itself. Here we describe an approach where the use of multiple techniques has enabled a complete physical characterization of the rubber glove material. This approach not only applies to nitrile gloves but can also be used for analysis of any rubber material, polymer coatings, thin elastomeric films, porous membranes or flat, flexible sheets.

1 Introduction



Figure 1: Powder free, nitrile gloves used for analysis. Standard glove thickness is 6 mil (152 μm).

Disposable rubber gloves are routinely used for maintaining personal hygiene, prevent contamination and enable the wearer to work safely in a toxic/hazardous environment. Rubber latex is a cross-linked polymer with a tensile strength in the 25-40 MPa range, with a typical elastic elongation of 800-900%.¹ Nitrile gloves are usually of lower tensile strength than latex but their elastic modulus is typically higher. Gloves can be ordered in a variety of different types, thicknesses, finished textures and colors. The global demand for gloves is estimated to be \$ 2.3 billion USD in 2020 alone and is expected to grow steadily throughout 2027.² Physical analysis of flexible materials like rubber and related materials is generally performed by tensile testing, IR spectroscopy and X-ray diffraction while surface characterization is performed by optical/fluorescence microscopy, SEM, TEM etc. The most important assessment of the quality of rubber gloves is their leakage or barrier properties to solvents, chemicals, and biological agents (bacteria, virus, pathogens).

It is well known that vinyl gloves are more susceptible to leakage of biological material (e.g. viruses) compared to nitrile or latex gloves.³⁻⁶

In this study, we demonstrate a new, holistic way to characterize surfaces of flexible rubber materials like nitrile gloves using a full length scale analysis approach. This approach makes use of multiple measurement techniques like gas physisorption (surface area and pore size down to the sub-nm scale), atomic force microscopy (AFM, μm -scale) and pycnometry for skeletal density measurement which also assesses down to the sub-nm level. This approach has the following benefits: a) provides a complete analysis picture of the material, b) demonstrates the use of multiple techniques (instruments) in the Anton Paar product portfolio and c) enables customers to use multiple techniques for getting a complete picture of the rubber glove surface and physical properties.

2 Experimental

Three different techniques were used in the characterization of nitrile gloves – gas pycnometry (Ultrapyc 5000), gas physisorption (Autosorb iQ) and atomic force microscopy (Tosca 400 AFM) to measure the skeletal density, rubber surface area and glove surface topography respectively. The results of these three measurements are summarized below in Table 1.

Results	Value	Technique used
Skeletal Density (g/cm^3)	1.09	Gas (N_2) Pycnometry
BET Surface area (m^2/g)	0.033	Gas (Kr) Physisorption
RMS Roughness (nm)	18.7	AFM
Average feature height (nm)	57.3	AFM

Table 1: Physical properties of nitrile rubber gloves as measured by three different surface characterization techniques.

For gas pycnometry, nitrogen gas was used for the measurement. For BET surface area via gas physisorption, Kr (77 K) was used. Kr (77 K) was necessary as an alternative to the more common nitrogen adsorption because of the extremely low surface area of the glove sample.⁷ Small pieces (~1-3 mm) of the gloves were cut and used for the above analyses.

For gas physisorption, the chopped glove pieces were used to fill a 9 mm large bulb sample cell of the Autosorb iQ system. Roughly 1.4 gm of the rubber glove was used for gas physisorption analysis.

For AFM measurements, the Tosca AFM was operated in tapping mode for capturing height and phase contrast of features. The outside glove surface was measured. The cantilevers used were Arrow-NCR10 silicon sensors from Anton Paar, with a nominal resonance frequency of 285 kHz and a force constant of 42 N/m. Cantilever dimensions were 160 µm in length and 45 µm in width. The image scan rate was 0.1 Hz, while scan resolution was set to 400. All measurements were done at ambient, room temperature conditions.

3 Results and Discussion

3.1 Gas pycnometry (Ultrapyc 5000)

Gas pycnometry measures the skeletal density of materials. A sample of known mass (weight) is introduced into a sealed chamber of known volume which is then pressurized with inert gas (nitrogen in the case of polymer samples such as gloves). Volume of the sample is then calculated from Boyle's law. Density is then calculated by dividing the mass by the sample volume. In this case, 0.8152 gm of chopped up nitrile glove was analyzed.

Results

True Density

1.0954 g/cm³

Average Volume

0.744 cm³

Percent Variance

0.0217 %

Figure 2: Skeletal density measurement of nitrile glove sample measured with the Ultrapyc 5000.

3.2 Gas physisorption (Autosorb iQ MP)

This technique is used to measure surface area of materials. Typically nitrogen gas is used as a "probe" molecule to adsorb into the pores of the materials either as a single layer (BET isotherm) or stacked,

multiple layers at a constant temperature (77 K for nitrogen). For low surface area materials, krypton is used instead of nitrogen due to its enhanced sensitivity.⁷ A complete explanation of BET theory can be found elsewhere.⁸ Materials having high porosity, typically also have high (> 10 m²/g) surface area values. BET surface area was measured twice on two different nitrile glove samples and the average was found to be 0.033 m²/g. This very low value is expected for non-porous materials like nitrile rubber glove material.

3.3 Atomic force microscopy (AFM) of glove surface

AFM allows characterization of surface morphology, roughness, grain size and presence of impurities or inhomogeneity's. The surface topography of the nitrile glove exterior surface is shown in Figure 3. Surface morphology is important for knowing properties of the glove material like grip and the wearer's comfort/feel.

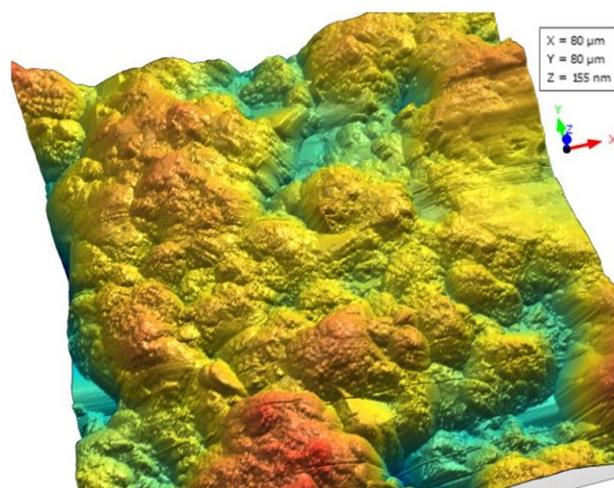


Figure 3: An 80x80 µm 3-D topography (forward trace) AFM image of the nitrile glove surface.

Example of an AFM (operated in tapping mode) image used for checking the homogeneity of the rubber film is shown below in Figure 4. Figure 4a) is the topography image of a 5x5 µm region showing no particular contrast. However the phase image of the same region shows clear contrast as shown in Figure 4b). This is a phase shift image of the cantilever as it tapped the surface, darker regions mean lower phase shift of the cantilever suggesting regions of softer material. The image shows regions of different constituent material – rubber Vs possibly the filler or pigment used. Thus the AFM phase image allows visualization of material differences in the rubber glove material.

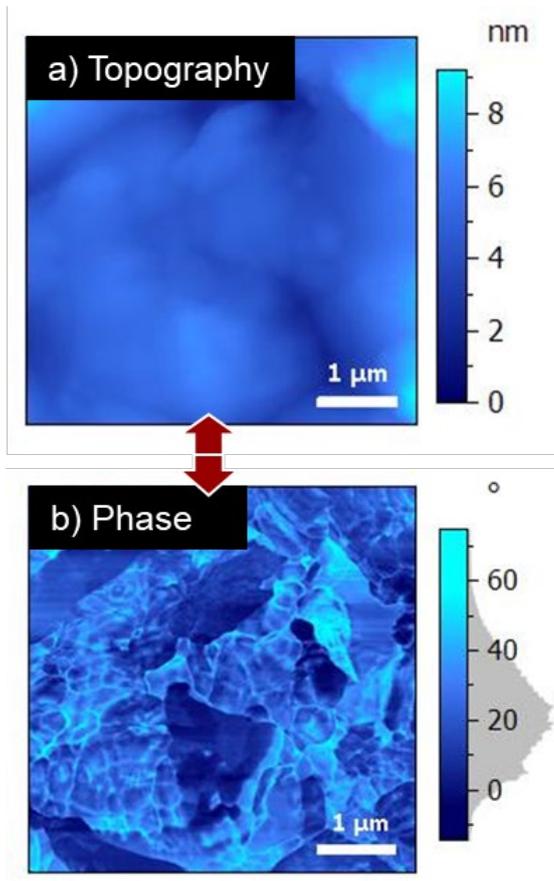
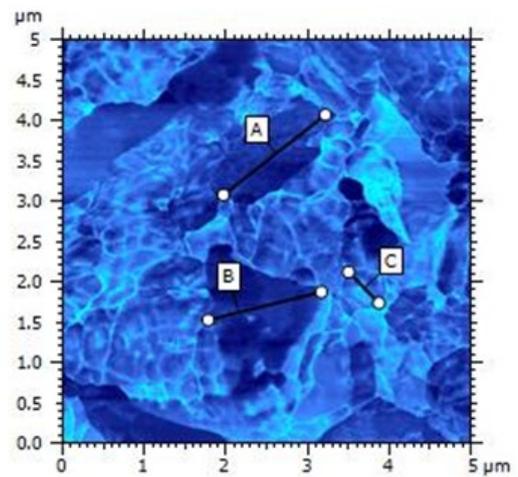


Figure 4: a) 5x5 µm AFM topography (forward trace) image of the glove surface showing no particular differentiation in materials. b) Phase (forward trace) image of the same region showing regions of different materials, possibly the filler or pigment added to the rubber.

Further, the AFM phase image shows clear grain size and boundaries of the different materials present as shown in Figure 5. Three different regions were measured – A,B,C. Regions A,B showed the same dark contrast while C was a different materials with a lighter contrast. A and B were also bigger (1.6 and 1.4 µm respectively) compared to C which was 0.53 µm. This grain size difference also confirms regions of different materials used during rubber glove manufacturing.



Distances	Unit	A	B	C
HDist	µm	1.6	1.4	0.53

Figure 5: Grain size measurements from the phase image showing differences in the regions of different contrast.

4 Summary

We have shown how three different surface characterization techniques – pycnometry, gas physisorption and AFM can work together to form a complete picture of the nitrile glove surface at various scales of measurement. Each technique has its own strengths and throws light on the glove surface, which can be used in various ways. Surface area and density values provide information which can be used for material characterization in the real world. For e.g. these values are measures of glove properties that influence wearer feel, diffusion/leakage of toxic materials and resistance to tear, etc. Changes in glove surface area have been shown to be a measure of rubber degradation.⁹ The AFM can show presence of non-uniformities, contamination and is useful for quality control in the glove material.

5 References

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