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Filter Autopsy and Analytical Techniques for Qualitative and Quantitative Comparison of Properties Before and After Testing and Due to Aging

Coralie B. Rose and Charles A. Waggoner

Institute for Clean Energy Technology, Mississippi State University, Starkville, MS

ABSTRACT

The Institute for Clean Energy Technology (ICET) at Mississippi State University has developed a full suite of analytical techniques for High Efficiency Particulate Air (HEPA) filter evaluation in concert with and in support of testing of filters and filter media. This analytical approach combines aspects of qualification testing with techniques that enable root cause determination for filter failure and prompts alternate filter and media development based on results from each autopsy. Media are evaluated using Scanning Electron Microscopy, Energy-Dispersive X-Ray Spectroscopy, Tensile Strength Determination, Compressibility and Recovery Determination, Thermal Analysis (TGA, DSC, DTA), and Gravimetric Mapping. Filter configuration is evaluated using Three Dimensional Imaging and measurement software. Three Dimensional Imaging and Analysis of filter pack geometry before and after testing reveals changes resulting from weakening of media or effects of increased differential pressure.

The methodology developed for filter autopsy and analysis incorporates a wide range of techniques and capabilities in filter evaluation that far exceed the current standard and that might be useful to both further product development and policy decisions regarding current use and life cycle.

INTRODUCTION

Current research activities underwritten by the United States Department of Energy (DOE) involve evaluating the effects of age and exposure to elevated conditions (temperature and relative humidity) on performance capabilities of ASME AG-1 nuclear grade HEPA filters. Studies at ICET have included moisture failure, source term loading, seal and pinhole leaks, and media velocity [1,2]. The design, construction, and operation of ICET test stands used in these research efforts have been communicated in various papers, proceedings, and conference discussions [3,4].
Research efforts that have been conducted for further evaluation of changes associated with filter testing and aging include loading pattern mapping, filter rupture or failure mapping, mechanical property loss location, and degradation of filter media components. This evaluative suite requires a methodical disassembly of loaded filters and a full characterization and parameter mapping. Pre- and posttest determined changes in the filter pack geometry or the filter media can be attributed to testing

stresses or to age. The autopsy examination (methodology) will indicate not only the existence of basic operational failure of filters or media in response to testing or aging, but ultimately may allow a determination of cause based on the results of each analytical investigation.

ICET employs a combination of analytical techniques to the autopsy that correlate to how the filter has been tested. The full compliment of techniques include the following:

- Three Dimensional Imaging of Configuration
- Thermal Analysis (TGA, DSC, DTA)
- Tensile Strength Determination
- Compressibility and Recovery Determination
- Scanning Electron Microscopy
- Energy-Dispersive X-Ray Spectroscopy

In addition to common filter evaluation techniques and standards from AG-1, ICET has developed a set of novel analytical techniques to further evaluate filter design and media and provide post testing investigation. A tailored set of analytical procedures is selected based on filter design type and testing conditions. Ultimately, this is intended to establish a platform for a causal analysis. Failure causes are probed based on the results of each analytical technique. The ability to determine the location of potential failure sites and sites of decreased functionality will be integral to design prototype testing.

Goals in filter autopsy are to:

- record configurational changes to pleats before and after testing
- provide mapping of mass loading throughout the filter pack
- classify loaded particle populations at specific sites across the full surface area of the filter pack
- determine mechanical properties of the media at various locations throughout the filter pack
- measure and map the filter cake thickness as a function of location and orientation in the test stand housing or location relative to the seam
- determine a quantitative measure of the degradation of fiber coatings within filter media to support and explain any loss of mechanical properties.

ANALYTICAL TECHNIQUES

Three Dimensional Configuration Comparison for Pleat Distortion

Testing activities can lead to filter failure and can have clear configurational effects. Examples of configurational changes to a filter pack are: pleat rupture with an associated offset distance, as shown in Figure 1 left and right; distortion of pleat geometry such as ballooning or blowout as shown in Figure 2 right and compared to typical configuration in Figure 2 left; and pleat dislocation, or media buckling, when a pleat edge changes orientation and tilts past the angle of normal to the top and bottom, shown in Figure 3 left and right.

Figure 1. Photos of pleat rupture (left) with offset distance (right)

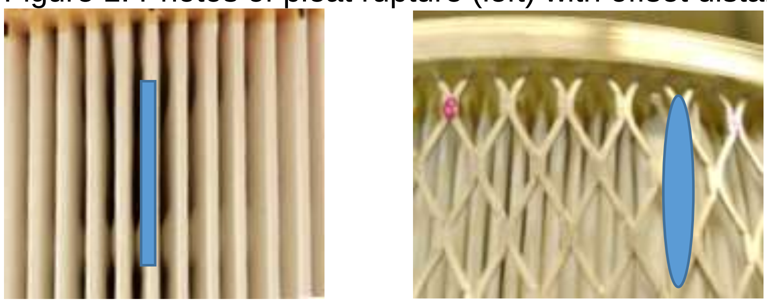


Figure 2. Photos of typical pleat geometry (left) and ballooned pleats (right)

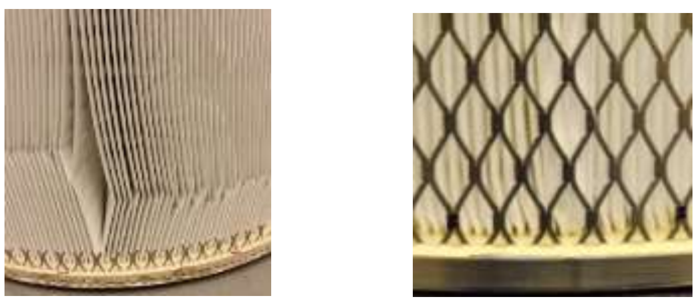


Figure 3. Photos of pleat creasing and stress (left) and buckling of media (right)

Pleat disruptions can in this way be quantitatively recorded using a three dimensional laser imaging system. The configuration scan prior to testing sets up an initial baseline for the pleat geometry before testing. The scanning system is shown in Figure 4 for radial flow (left) and axial flow filters (right).

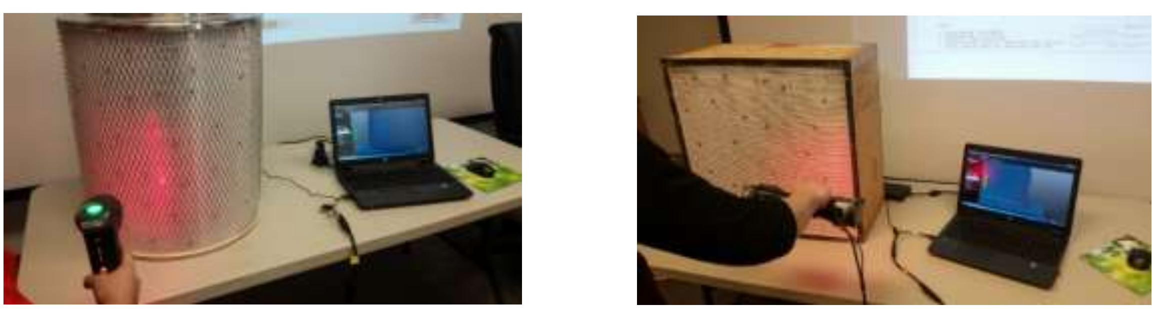


Figure 4. Photos of the three dimensional laser scanning process for a radial (left) and an axial flow filter (right)

All pleat edges are recorded in a three dimensional software model with a 0.2 mm resolution. Each pleat edge is measured using the inspection software in order to catalog the pleat geometry prior to testing. The surface area of the pleat edge is calculated from measurements taken in VXinspect software. Once testing is complete, a second three dimensional scan is performed and the pleat edge geometry is once again measured. A catalog of pleat edge geometry is taken for the final scan and the two catalogs are compared statistically in order to determine differences in geometry. Also, VXinspect software has the capability to compare the two models and generate a best fit alignment with a color map, where the color scale can also be equipped for or modified with the error distribution. These two methods will be used to report comparison results between before and after testing configurations and allow identification of configurational differences in the filter as a result of testing activities.

The laser scanning system used is Creaform Handy Scan 700, which can achieve 0.2 mm resolution and perform a quantitative comparison of two models. The software developed by Creaform is VXelements; a three dimensional image provided for measurement and inspection is shown in Figure 5 and a comparison of pleat geometry is shown in Figure 6.

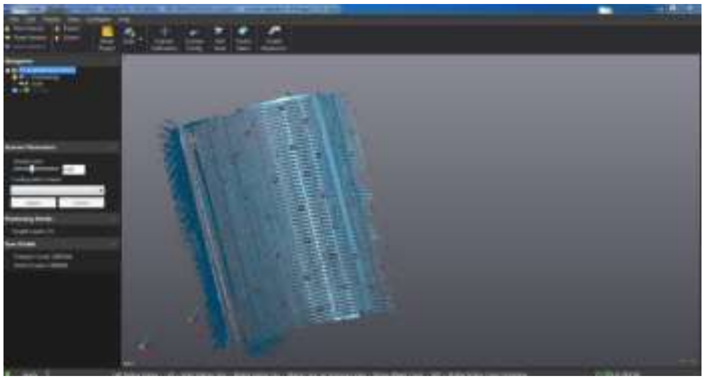


Figure 5. Radial flow filter three dimensional image

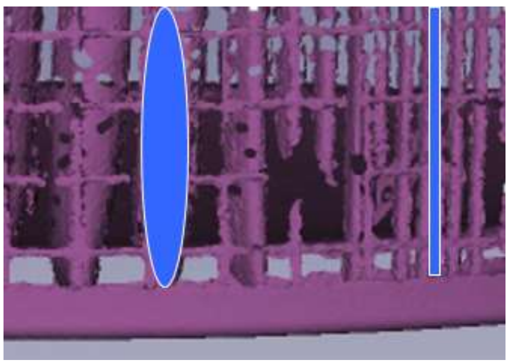


Figure 6. Comparison of pleat geometry using three dimensional imaging software

Thermal Analysis

Thermogravimetric analysis (TGA), Differential Scanning Calorimetry (DSC), and Differential Thermal Analysis (DTA) provide a quantitative measure of coating degradation for each component of the filter media and information on heat flow and thermal properties. Degradation of the acrylic binder on filter fibers has previously been correlated with decreased tensile strength of the media [5] and is an important factor in life cycle limitations in filters.

Acrylic binder used in most filter media to impart mechanical strength is removed from the sample at 250-480 °C. The delta Y is calculated to determine the composition of the media and the weight percent of binder. In this way, an initial themograph is generated and compared to a final thermograph of aged or tested media. Since the coating weight is roughly 1-4% of the sample weight, an instrument with a high balance resolution and sensitivity is required to determine small differences within this 1-4% weight range.

The instrument and method used is Pyris-Diamond TG/DTA 6300, shown in Figure 7; Ramp 10 °C/min to 1000 °C, hold 5 minutes at 1000 °C under a N_2 flow rate of 20 mL/min using a platinum pan and alumina reference. Also in use is a TA Instruments SDT Q600 simultaneous analyzer at the same method and conditions.

Figure 7. Simultaneous analyzer Pyris-Diamond TG/DTA 6300

Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) allows characterization of particle population by morphology and particle size. Figure 1. shows a sample of filter media from a specific manufacturer loaded with propane particles. The fiber coatings can be seen in this image as the webbing on each fiber. Penetration depth and filter cake thickness can also be determined using SEM, as shown in Figures 14 and 15. The JEOL 6500F Field Emission SEM with a resolution of 5 nm and a 5 kV accelerating voltage is used to image filter media at sites of rupture and throughout the surface of the filter pack in order to expose loading patterns and report the size and morphology of the particle population.

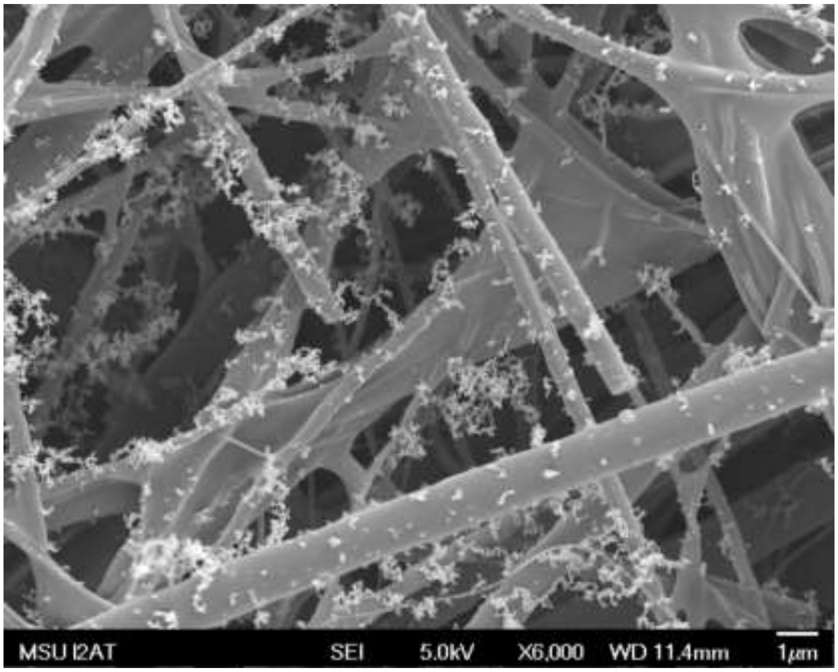


Figure 8. Scanning Electron Micrograph of Propane-Loaded Filter Media

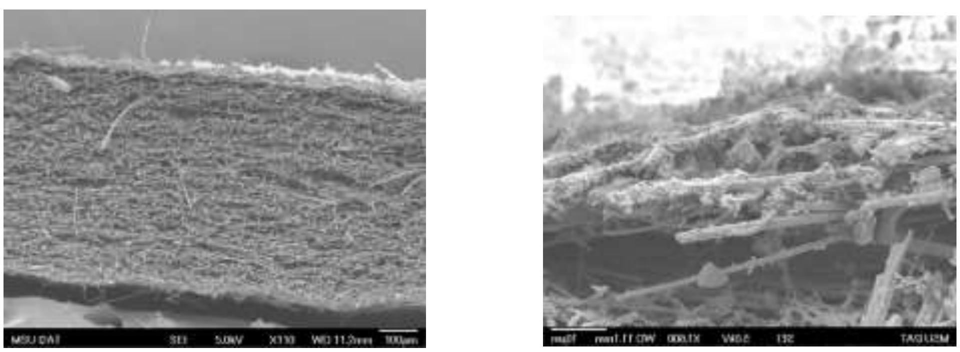


Figure 9. SE micrographs of the filter cake (left) and a rupture edge (right)

Applicable standards are ASTM E1617-09 (14) Standard Practice for Reporting Particle Size Characterization Data and ASTM F1877-05 (10) Standard Practice for Characterization of Particles [6,7], used for providing and reporting data on morphology.

Energy-Dispersive X-Ray Spectroscopy

Energy Dispersive X-Ray Spectroscopy provides elemental mapping for a further measure of coating degradation and loading location. A Zeiss EVO 50VP Variable Pressure Scanning Electron Microscope provides an elemental mapping for specified elements. In Figure 16, EDX was performed on an unloaded filter media sample to map oxygen. Figure 17 is the corresponding SEM image of unloaded filter media. A loaded sample was imaged using EDX to map sodium (Figure 18); Figure 19 is the same image without the specialized mapping in order to allow identification of structures within the EDX images. An example of an EDX spectrum for loaded filter media is shown in Figure 20.

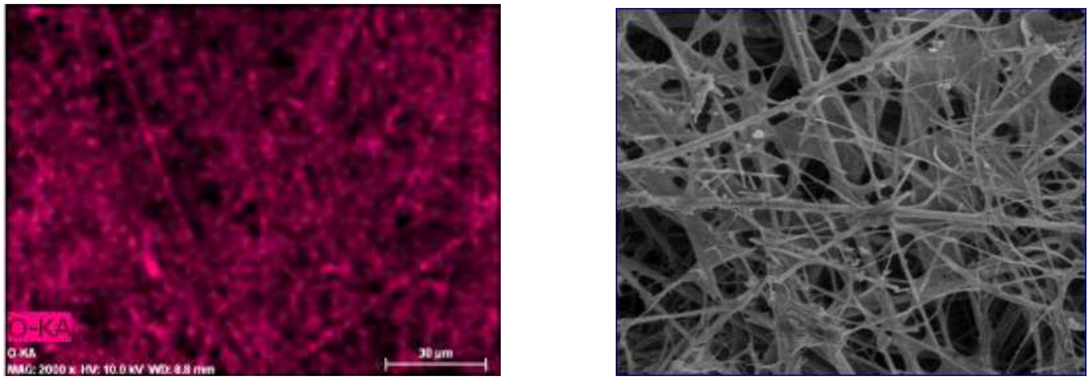


Figure10. EDX Elemental Map for Oxygen (left) and corresponding SEM (right)

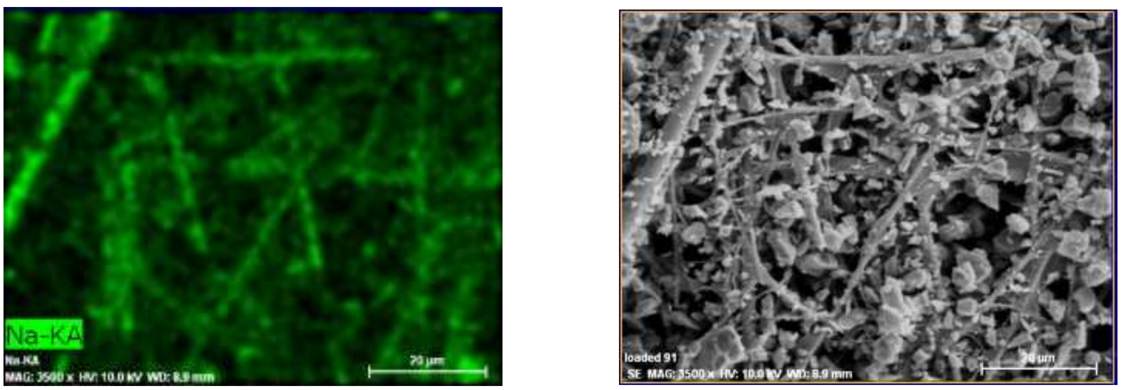


Figure 11. EDX Elemental Map for Sodium (left) and corresponding SEM (right)

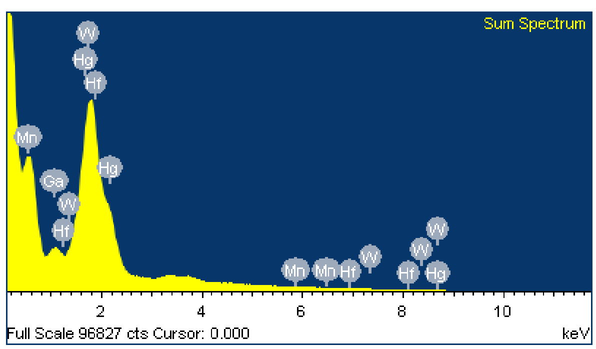


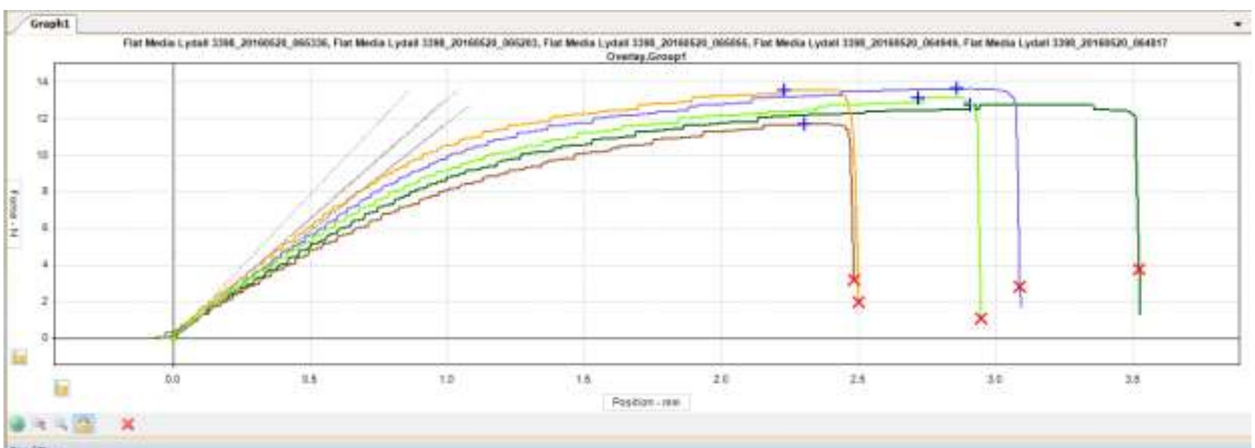
Figure 12. EDX spectrum results for loaded filter media

Tensile Strength Determination

Tensile strength tests of the filter media have been used to identify filters with degraded strength and potential for operational failure. Identification of reduced mechanical properties of filter media is performed using a Thwing-Albert Vantage NX Universal Tester with pneumatic grips. Testing is shown in Figure 21. The standard for TAPPI/ANSI T 494-om13 Tensile Properties of paper and paperboard (using constant rate of elongation apparatus), along with TAPPI T402-sp13 Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products [8,9] have been followed, including pretreatment of samples and environmental controls.

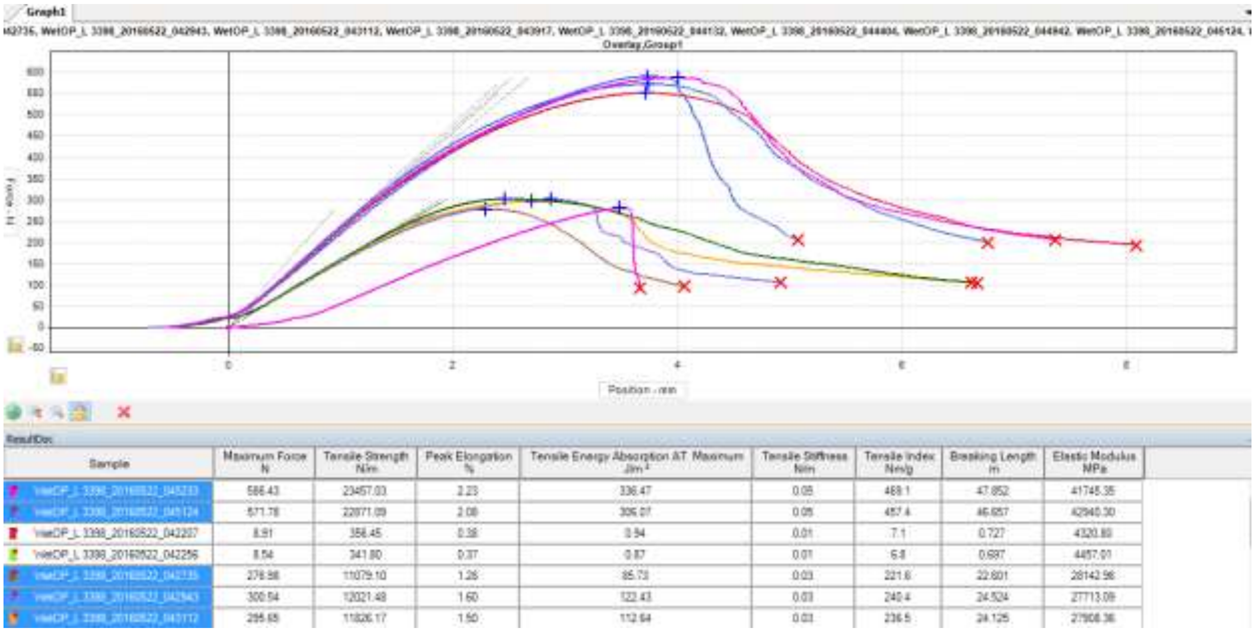
Figure 13. Tensile strength testing of filter media sample

The results of flat media tensile testing with preconditioning are shown in Figure 14. A wet overpressure simulation was performed with media from one manufacturer using no scrim, single scrim, and double scrim media samples, as shown in Figure 15. These sheets were exposed to conditions of 35 ±3 °C and 95 ±5 % relative humidity for 24 hours [10], then preconditioned according to the TAPPI standard. Samples were then cut from the sheets, the masses were taken, and half were placed to soak in deionized water for one hour, while the rest were spritzed with water and conditioned for another hour, after which a final mass was taken and testing was performed.



Sample	Maximum Force N	Tensile Strength N/m	Peak Elongation %	Tensile Energy Absorption AT Maximum J/m²	Tensile Stiffness N/m	Tensile Index Nm/g	Breaking Length m	Elastic Modulus MPa
Flat Media Lydall 3398_20160520_064817	13.06	522.46	1.51	5.57	0.00	10.4	1.066	2358.93
Flat Media Lydall 3398_20160520_064949	11.66	466.31	1.28	3.97	0.00	9.3	0.951	2123.55
Flat Media Lydall 3398_20160520_065556	13.61	544.43	1.59	6.33	0.00	10.9	1.111	2342.06
Flat Media Lydall 3398_20160520_065203	13.49	539.56	1.24	4.67	0.00	10.8	1.101	2824.09
Flat Media Lydall 3398_20160520_065336	12.70	507.81	1.62	5.87	0.00	10.2	1.036	2107.33
Wet Overpressure Sim: Undefined								
Average	12.90	516.11	1.45	5.28	0.00	10.3	1.053	2351.19

Figure 14. Tensile Strength Testing of Dry Filter Media



Sample	Maximum Force N	Tensile Strength N/m	Peak Elongation %	Tensile Energy Absorption AT Maximum J/m²	Tensile Stiffness N/m	Tensile Index Nm/g	Breaking Length m	Elastic Modulus MPa
WetOP_L 3398_20160522_045233	586.43	23457.03	2.23	336.47	0.05	469.1	47.852	41745.35
WetOP_L 3398_20160522_045124	571.78	22871.09	2.08	306.07	0.05	457.4	46.657	42940.30
WetOP_L 3398_20160522_042207	8.91	356.45	0.38	0.94	0.01	7.1	0.727	4320.80
WetOP_L 3398_20160522_042256	8.54	341.80	0.37	0.87	0.01	6.8	0.697	4457.01
WetOP_L 3398_20160522_042735	276.98	11079.10	1.26	85.73	0.03	221.6	22.601	28142.98
WetOP_L 3398_20160522_042943	300.54	12021.48	1.60	122.43	0.03	240.4	24.524	27713.09
WetOP_L 3398_20160522_043112	295.65	11826.17	1.50	112.64	0.03	236.5	24.125	27908.36

Figure 15. Tensile testing results of soaked versus wetted filter media (wet overpressure test media simulation)

Compressibility and Recovery Determination

Compressibility and Recovery of Filter Media samples have been expected to change as a factor of changing differential pressure. The testing standard used for this compressibility determination is ASTM F36-15 Standard Test Method for Compressibility and Recovery of Gasket Materials [11] and testing is performed using a Thwing-Albert Vantage NX Universal Tester with compression apparatus, shown in Figure 16.

Figure 16. Compressibility and recovery testing for filter media

Goals in Filter Autopsy

In order to provide details of changes due to testing activities or performance effects from aging, the filter autopsy method must preserve all changes that occurred during testing and must not disturb the filter media pack. The disassembly method must be as noninvasive as possible to limit or minimize vibrations that can cause dislodgement of particle loading. Physical stresses must also be curtailed to prevent distortion of pleat geometry and resulting alteration of mechanical strength testing results. As such, a method has been developed that minimizes movement of the filter and reduces excessive vibration. Both mechanical and thermal methods for disassembly have been developed.

Process of Autopsy and Analysis

Autopsy Procedures are performed as illustrated in the flowchart, Figure 17.

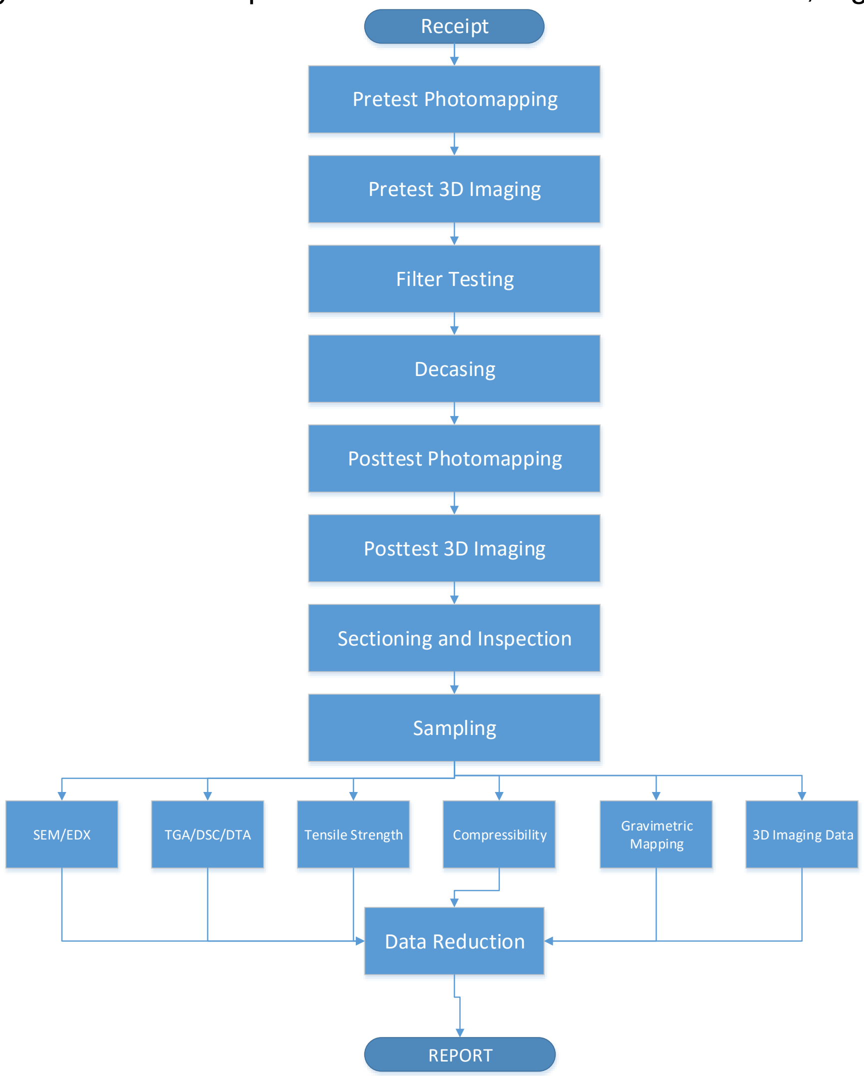


Figure 17. Flowchart of Procedures for Autopsy and Analysis

Pretest data is collected by photomapping and Three Dimensional Imaging. An initial configuration for the filter pack is measured using the software and recorded. The filter is provided for testing activities. Once testing is complete, decasing and disassembly are performed. The filter is then photomapped and 3D imaged for posttesting configuration elucidation. Sectioning and Inspection allows notation of areas of interest within the filter pack, and sampling is performed over the full surface area of the filter pack. Analytical procedures are performed using provided non-tested flat sheet media of the same media lot for initial properties characterization, and all excised tested samples are prepared for analysis. Analysis is performed and all data is compiled and analyzed, then reported.

Decasing Method

Filter autopsy provides the capability to perform coating degradation determination, tensile strength and compressibility location determination, mass loading mapping, and to examine filter properties before and after testing by providing access to the interior for sampling and analysis. Integral to the analytical efforts in filter evaluation has been the strategy of decasing the filter pack to provide access for analysis without biasing analytical results by disturbing particle loading, causing stress or strain to the filter media, and distorting the tested pleat geometry.

An angle grinder is used to remove the vertical support bars and endcap lip for radial flow filters for both top and bottom of the filter with a minimal vibrational effect, as shown in Figure 18 left. Safe change and remote change filters have different removal considerations. For a safe change filter, the gasket channel is first removed by carefully drilling out the channel spot welds, as shown in Figure 18 right.

Figure 18. Photos of removal of endcap lip (left) and removal of channel (right)

A channel barrier is created to exclude the filter pack, the filter is leveled and liquid nitrogen is poured onto the endcap, as shown in Figure 19. The nitrogen is allowed to boil off for 5 minutes and the endcap is removed using a pry tool.

Figure 19. Photos of application of liquid nitrogen (left) and boiling off of liquid nitrogen (right)

Once both endcaps have been removed, the protective grill is cut using bolt cutters and removed to expose the filter pack and mastic, as shown in Figure 20 left. The decased radial filter is then sectioned into 15 sections using a heated blade knife, while axial flow filters are sectioned into 3 sections. Axial flow filters must be treated uniquely for each manufacturer design. Removal of bolts, rivets, or nails is required for decasing and weld spots are removed to expose the filter pack. The full range of designs are displayed in Figure 21.

Figure 20. Photos of removal of protective grill (left) and removal of an axial flow filter side casing (right)

Figure 21. Photos of axial flow filters design differences; rivets (left), bolts (center), and nails (right)

Sampling

Simple random sampling is performed for each region of a section over the full surface area of the filter pack. A random number generator in Microsoft Excel is used to provide the representative sampling sites for each technique. Sampling regions are illustrated in Figure 22, while the random number generation is shown in Figure 23.

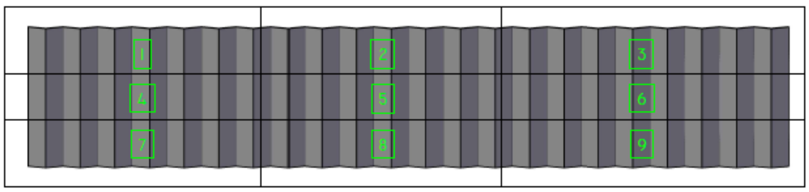
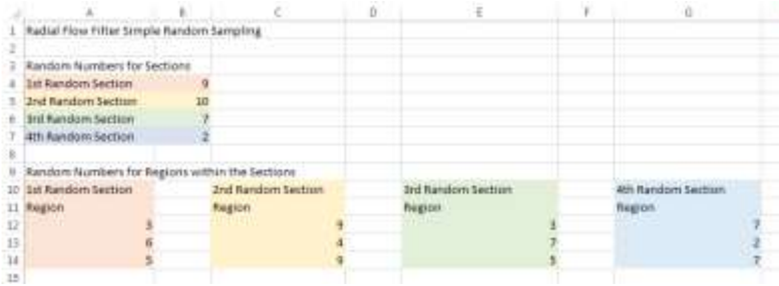


Figure 22. Sampling regions of a filter section



Radial Flow Filter Simple Random Sampling				
Random Numbers for Sections				
1st Random Section	9			
2nd Random Section	10			
3rd Random Section	7			
4th Random Section	2			
Random Numbers for Regions within the Sections				
1st Random Section		2nd Random Section	3rd Random Section	4th Random Section
Region		Region	Region	Region
3		9	3	7
6		4	7	2
5		9	5	7

Figure 23. Simple random number generation for sampling within each section

As evaluated by J.K. Frethold [12], sampling types are based on mechanisms of filter failure from observed field events. Tensile strength testing of the media in the machine and cross directions are taken, for both the pleat face solely and also for samples taken across the fold. Military specifications, ASTM and TAPPI standards, and literature reported techniques require tensile strength samples to be 25.4 x 152.4 mm (1x6 inches). Because radial flow filters have a pleat face of generally only 76.2 mm (3 inches), radial filters have two types of machine and cross direction sample dimensions. The 76.2 mm samples in both machine and cross directions and folded and face compositions are compared to the standard length samples 152.4 mm of the same width. Types of samples harvested are illustrated in Figure 24 left and shown after excision in the image on the right.

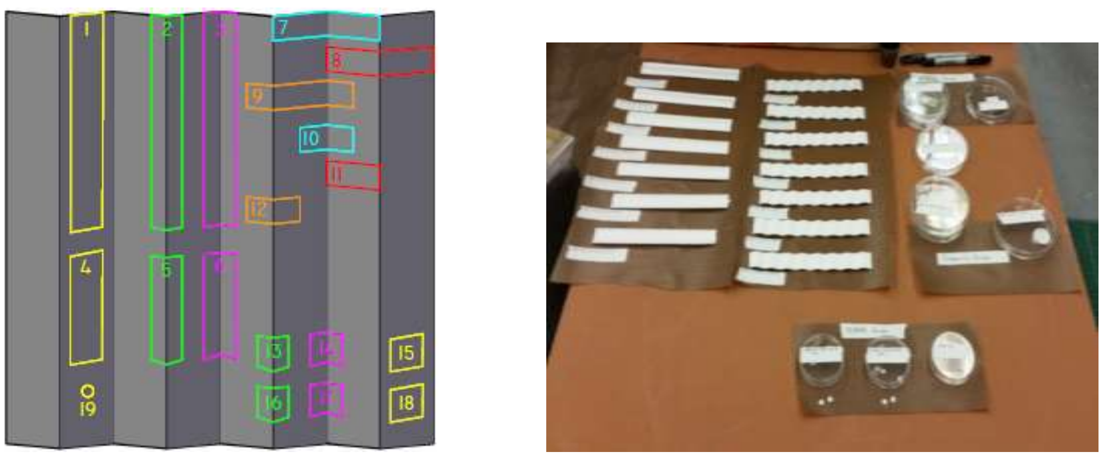


Figure 24. Images of types of samples for analysis (left) and samples ready for analysis (right)

From Figure 24 left, sample types 1-3 are all cross direction typical length tensile strength samples and types 7-9 are corresponding machine direction typical length samples, while sample types 4-6 are cross direction short length samples and types 10-12 are machine direction short length samples. Sample types 13-15 are samples for SEM analysis, and sample types 16-18 are compressibility determination samples. TGA samples are represented as sample type 19.

In addition to representative sampling of each region, care is taken to visually inspect the full filter pack and locate ruptures or distortions in the pleat geometry or the media. Samples are taken from edges of rupture sites for later SEM analysis. Figure 25 illustrates the transection of each section of a radial flow filter (left) and a typical inspection and cataloging of each site of rupture is shown at right.

Figure 25. Photos of transects of a section (left) and inspection of rupture sites (right)

Sampling will also be performed for radial flow filters relative to the seam and to the orientation in the test stand in order to detemine flow and loading conditions inside the annulus. Figure 26 shows an axial flow filter sampling pattern for one pleat.


Figure 26. Sampled pleats in axial flow with (left) and without separators (right)

RESULTS

Statistical Treatment of Data

Pretest and posttest data sets from each technique are compared using the non-pooled t test and a full statistical analysis is performed in Microsoft Excel. Figures 27 and 28 show all methods and the impact of each technique on property characterization.

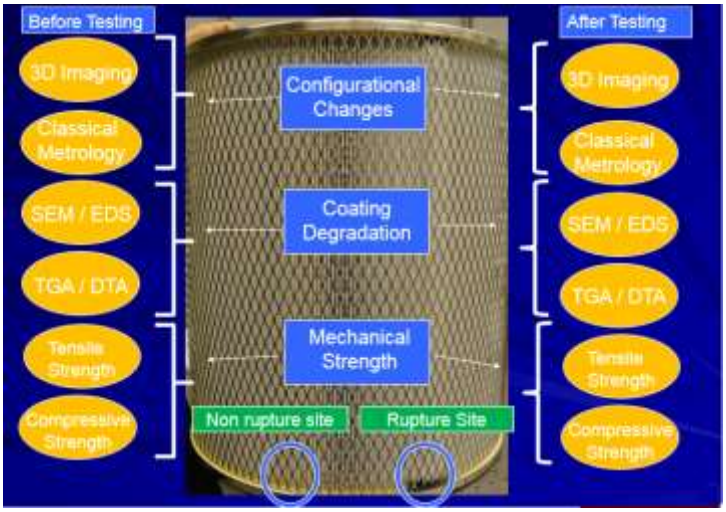


Figure 27. Filter and filter media characterized before and again after testing

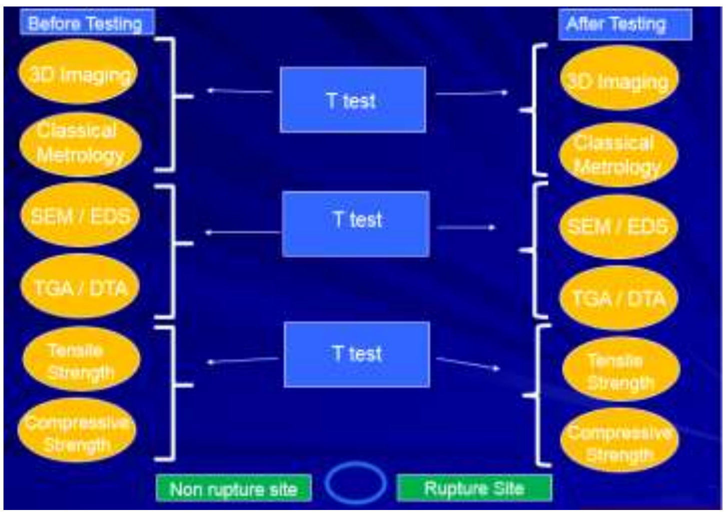


Figure 28. Non-pooled t-test to determine significance of differences in the before and after data sets

DISCUSSION

The performance of filters throughout testing can be gauged by our unique analytical autopsy approach to identifying parameters active in filter performance and as causes of degradation of function. Structural damage and configurational changes caused by testing activities are cataloged and quantified, while a full property characterization is made of the filter before and again after testing. Comparison of the data will allow identification of issues in filter performance and help to identify trends.

Among the current data compiled with our array of techniques are, tensile strength of the medium as a function of location, compressibility and recovery as a function of location, compositional change provided by thermogravimetric analysis, three dimensional configurational comparison and location of distortions in pleat geometry, and particle population as a function of location with SEM and EDX imaging. Criteria taken from these results will inform regarding operational failure in HEPA filters.

Future Work

Future work will include addition of several analytical techniques and autopsy of a nontested filter for determination of mass variability of clean media throughout the full surface area of the filter. In this way the deposition will not only be able to be mapped, but the compressibility and loading can be seen as a function of the type of housing used for testing.

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