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"Sample and/or Specimen Preparation for Testing Multilayer Geomembranes"

by

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Introduction

The identification of individual components of a multilayer geomembrane is proving to be quite difficult. From a forensic sense, such a procedure is often called "fingerprinting". By a multilayer geomembrane we mean one that is composed of more than a single horizontal layer of material within its total thickness. This White Paper is focused on sample and/or specimen preparation for the subsequent testing of multilayer geomembranes where the individual layers have chemical bonds between them.

It must be recognized at the outset that all polymeric geomembranes are actually formulations of a parent resin from which they derive their generic name. The most commonly used geomembranes for liquid and gas barriers are polyethylene (PE), flexible polypropylene(fPP), polyvinyl chloride (PVC), chlorosulfonated polyethylene (CSPE) and ethylene propylene diene terpolymer (EPDM).

There are three common ways to manufacture geomembranes; i.e., extrusion, calendering and spread coating. These processes are fully described in many publications including Schiers (2009), Müller (2007), Koerner (2012) and others. The different processes allow for considerable variability in the end product. For the conventional type of geomembranes that are uniform throughout their thicknesses, they consist of polymeric resins mixed with additives, such as carbon black and antioxidants, as required for their processibility and performance. *As such, the entire thickness of the typical geomembrane is of the same formulation.* This type of geomembrane has provided the large majority of polymeric barriers to liquids and gases to date.

Currently, all have well established generic specifications available via the GSI website at http://www.geosynthetic-institute.org/specs.htm and/or others.

Within the past ten years, however, multilayer geomembranes consisting of chemically bonded discrete layers of different resins and/or different formulations have become available. They are manufactured by a process called "coextrusion" whereby several streams of material meet to form the final layered product. Figure 1 shows the process for blown film extrusion of a three layered geomembrane. Flat die manufacturing similarly produces multilayer geomembranes, with separate dies providing each layer as they exit their respective extruders in a horizontal, rather than circular, configuration. The individual layers are indeed chemically bonded together. Such products include, but are not limited to, the following:

- single or double sided textured layers
- HDPE/LLDPE/HDPE composites
- vapor barrier inclusions
- tie layers between components
- layer(s) of different colors
- electric conductive layers

Note that scrim reinforced geomembranes are indeed multilayered but their manufacturing is generally done by lamination of already formed plys onto a fabric substrate or superstrate. The individual plys are relatively easy to separate and analyze individually. They will not be discussed herein.

The purpose for the differing layers in a chemically bonded multilayer geomembrane is to either provide new properties, enhance existing properties, change the appearance, or reduce the cost. The possibilities are endless and there is indeed a large range of options. See Figure 2

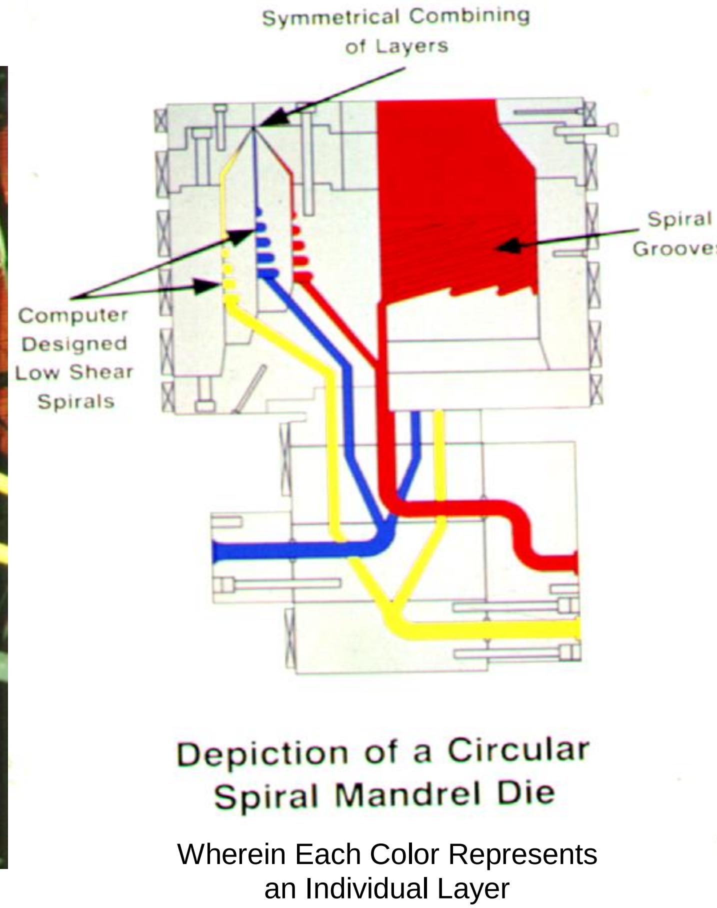
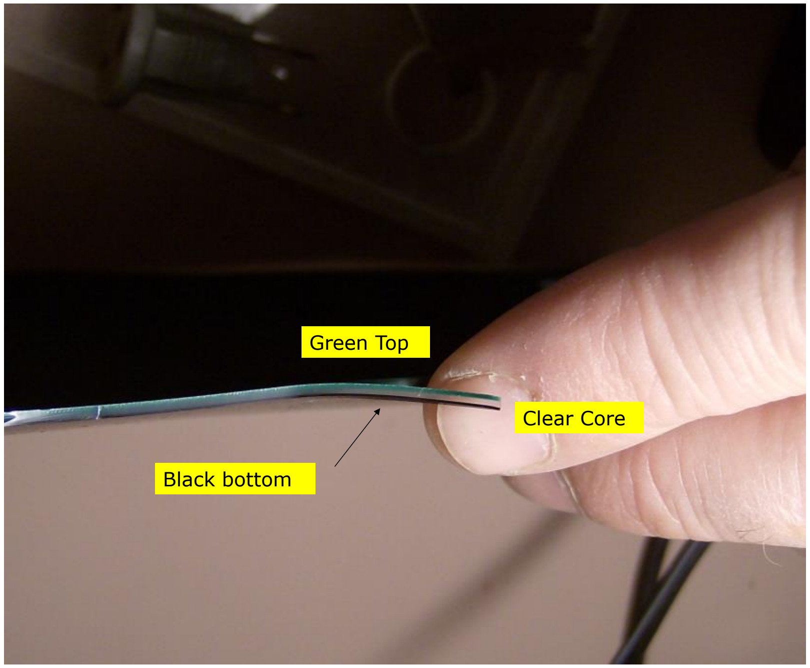
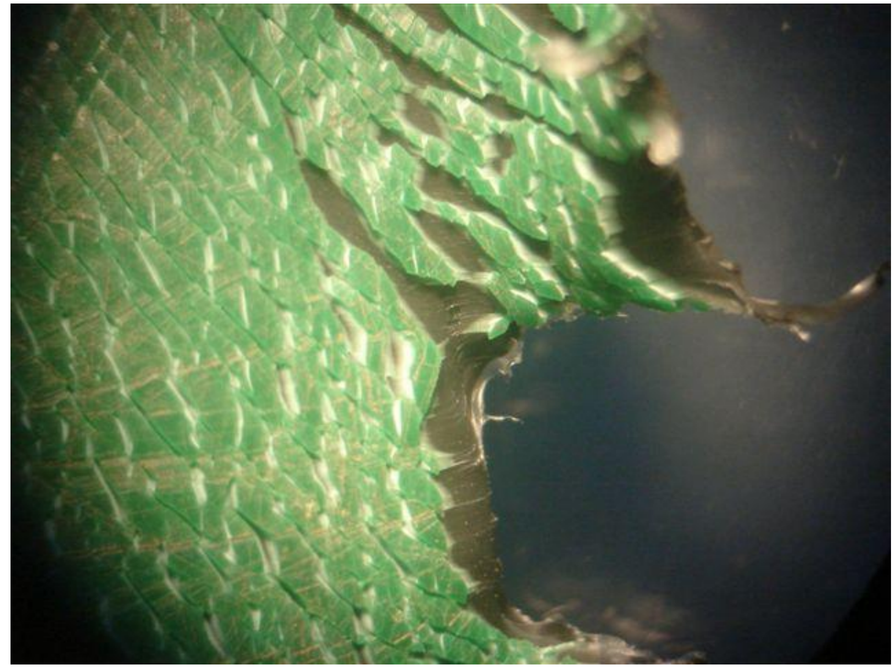


Figure 1. Blown film extrusion of a multilayered geomembrane.
(compl. GSE Lining Technology, Inc.)



(a) Example of three layers in a multilayed geomembrane



(b) Uneven layer degradation of example shown

Figure 2. Multilayered geomembrane used for anaerobic digester for containment of biogas.
(comp. of M. Ossa)

for a three layered geomembrane and particularly its nonuniform degradation pattern. *The above said, this White Paper is focused on sample and/or specimen preparation in order to proceed with standard laboratory testing and evaluation of such multilayer geomembranes where chemical bonds are involved between each layer.*

With this particular type of multilayer geomembrane, the task of identifying such geomembranes in-toto or each individual layer for conformance to specifications has become a challenge. One can certainly take a cross section of the material through its entire thickness and perform most physical and mechanical tests but these are "average" values (called **"homogenized"** properties herein) which are often inadequate for a detailed investigation. Alternatively, if each component layer of a multilayer geomembrane is desired to be evaluated separately it will require **"dissection"** of the individual layers. In light of the above, sample/specimen preparation by homogenization and dissection will be considered separately in the discussion to follow.

Prior to investigating any of these multilayered geomembranes, however, there needs to be an open and frank discussion between the parties involved as to what should be tested, how coupons or samples are obtained and how test specimens should be prepared prior to proceeding into a testing program.

Homogenization

If one desires the average value of a complete multilayer geomembrane, like density or tensile strength, a test specimen can be taken directly through the entire sample's cross section and evaluated in a standard manner. In such a way, the various layers are inherently averaged together. This is the simplest and most straightforward manner to proceed but does nothing to address the individual contributions of each specific layer. For some homogenized properties, however, the material must be ground or milled and then used as a homogenized powder, or the powder can be reconstituted into a compression molding plaque and sampled accordingly. Cryogenic grinding or milling is the act of cooling a material and then reducing it into a small particle size. In this regard, thermoplastics are difficult to grind into small particle sizes at ambient temperatures because they soften, adhere in lumpy masses, and clog screens. When chilled by dry ice, liquid carbon dioxide or liquid nitrogen, however, the thermoplastics can be finely ground into powders directly suitable for some tests. As seen in Figure 3, these mills are sophisticated and upon processing, should not change the behavior, fingerprint or structure of the material. For some tests the subsequent powder can be used directly. See Table 1.

Figure 3. Examples of cryogenic grinding mills. (ref. Google)

Table 1. Applicable Test Methods for Powders Obtained from Multilayer Geomembranes

Test	ASTM Designation	Typical Specimen Mass
Density	D792/D1505	5 mg
Melt Index	D1238	20 g
C. B. Content	D4218	5 g
Std-OIT	D3895	3.5 mg
HP-OIT	D5885	3.5 mg

If, however, the testing requires average sheet material, the powder or small pieces of the multilayered material can be molded into a plaque. This is done in compression molding devices as seen in Figure 4. Compression molding is a method of molding representative material in an open and heated cavity. The mold is contained while heat and pressure are maintained until the molding material has melted and is then cured. The devices employ feedback on pressure and temperature to control the consistency and the amount of flashing. Depending on the size of the resulting plaques, it is possible to perform essentially all physical and mechanical tests that are customary to geomembrane testing protocols.

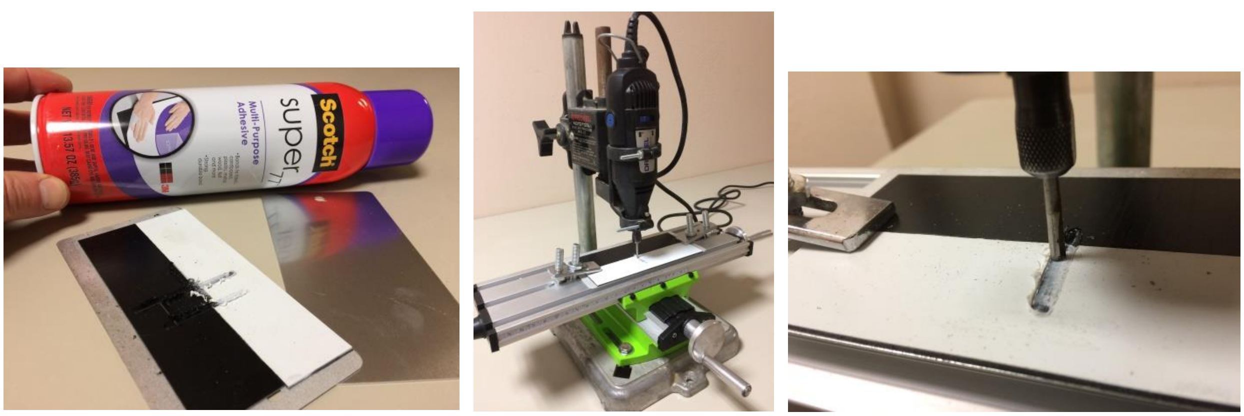
Figure 4a. Small sample compression molding device. (ref. Google)

Figure 4b. Large sample compression molding device. (ref. GSI)

Dissection

Converse to evaluating a homogenized powder or a reconstituted plaque, the multilayer geomembrane can be separated into its individual layers. That said, the multilayer products mentioned in the introduction cannot be easily separated (due to their chemical bonding) and there needs to be a method to carefully isolate individual layers of the cross section. Three options for such dissection include milling on an X-Y table, cutting with a sharp blade, or straining the composite material and then separating the debonded layers.

“Milling” is a machining process of using rotary cutters to remove material from a workpiece (e.g., a geomembrane mounted to a rigid substrate with contact adhesive) by advancing the specimen in a direction and at an angle with the axis of the tool which is rotating. It covers a wide variety of different operations and types of attachments. It is a well-known process and can be performed on a small scale; see Figures 5 (a to c). For multilayer geomembranes, one is interested in the filings, i.e., the scrap or waste being trimmed away from the geomembrane, and not on the remaining coherent piece mounted to the X-Y table. Note that the filings need to be collected and kept free of contamination if they are to be used as a test sample or specimen. The technique is precise and safe but requires patience. With a variable speed rotary tool, the cutting speed can be controlled so that little heat can be generated during the process. This technique is equally important for precisely removing superstrate, substrate or internal layers. With the X-Y table horizontally leveled, a specific layer can be removed precisely to any given depth. Thus, individual layers can be subsequently removed, collected and utilized as a powder or compression molded into a plaque as described previously.



(a) Gluing sample to flat substrate (b) Milling machine on X-Y table (c) Rotary cutting of a layer of desired material

Figure 5. Milling of individual layers of mutilayered geomembrane. (GSI photos)

An option to milling includes the **"shaving"** of an individual layer off of the material with a sharp blade scalpel or Exacto® knife. When larger pieces of material are needed, a cheese cutter or sharp plane can be used for dissecting the components from one another. This procedure is seen in Figure 6. Note that it is necessary to work with full thickness strips of the multilayered geomembrane. These strips are taped to a rigid flat surface so that they can be shaved cleanly. This procedure is fast, clean and generates no heat which might affect the morphology of the material. Again, the removed shavings can be used by themselves or compression molded into a plaque depending on the test method used.

Figure 6. Small plane removal of white layer from multilayered geomembrane. (GSI photo)

Figure 7. Planar separation of layers after test specimen straining. (GSI photo)

A third method on how to separate multilayer geomembrane layers is to **"strain"** the complete multilayer geomembrane in a tension testing device. Individual layers usually have very different strain responses. Hence, as the sample is being strained in a CRE tension device the chemical bonds holding the different layers together may break individually and thus relinquish the component(s) in isolation. This will be self-evident when the material is strained rapidly. See Figure 7 in which a dogbone tensile test separated in-plane resulted in obtaining the two individual layers.

Summary

The laboratory testing of multilayer geomembranes represents a difficult challenge insofar as identification of the homogeneous geomembrane and/or the separate properties of each layer of the composite material. *This White Paper has focused on evaluation of both the homogeneous and dissected materials insofar as test sample and/or specimen preparation is concerned.*

For the homogeneous, albeit layered, material, some tests can be performed on the composite material as manufactured. For others, however, the material will have to be a fully blended material. In this regard, a cryogenic process of generating small pieces (or even a powder) is described. Such powder can be used directly but can also be processed into a uniform sheet by making a compression molded plaque. It can then be used as would the original material but now in a fully homogenized state.

Alternatively, and with some degree of difficulty, the multilayer geomembrane can be separated into its individual layers. Three methods are described; milling, shaving and straining. Each method is tedious and must be done with great care so as not to have cross-mixing of the individual layers during sample or specimen preparation.

References

ASTM D1505, Standard Test Method for Density of Plastics by the Density Gradient Technique

ASTM D3895, Standard Test Method for Oxidative-Induction Time of Polyolefins by Different Scanning Calorimetry

ASTM D4218, Standard Test Method for Determination of Carbon Black Content in Polyethylene Compounds by the Muffle-Furnace Method

ASTM D5885, Standard Test Method for Oxidative Induction Time of Polyolefin Geosynthetics by High-Pressure Differential Scanning Calorimetry

ASTM D7912, Standard Test Method for Density and Specific Gravity (Relative Density) of Plastics by Displacement

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