Qualitative and Quantitative X-Ray Diffraction Analysis for Forensic Examination of Duct Tapes-Preliminary Findings

Pressure Sensitive Tape Features

•Main components are a flexible backing and pressure sensitive adhesive (PSA)
•When applied to a surface, bonds immediately at room temperature with slight pressure
•Adhesive bonds with no physical or chemical change and with no more than slight pressure
•Adhesive bond can be broken without damage to the surface and leaving minimal residue

Types of Pressure Sensitive Tapes

•Polycoated cloth tape
•Vinyl tape
•Polypropylene packaging tape
•Saturated paper tape
•Office tape
•Cloth tape
•Filament/strapping tape

Duct Tape Backing

•Single or multiple layers of polyethylene in the range of 1.5 mm to 4 mm thickness
•Gray tapes commonly contain a small amount of aluminum to impart the silver color
•Inorganic materials such as talc may be added to improve water repellency and tear strength
•May exhibit characteristics imparted during the manufacturing process, such as calendering marks and striations.
•Lettering or designs may be on the surface or the underside of the polyethylene

Forensic Analysis of Duct Tape

•Examinations of physical characteristics
•Polarized light microscopy (PLM)
•Fourier transform infrared spectroscopy (FTIR)
•Pyrolysis gas chromatography (py-GC)
•Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS)
•X-ray fluorescence spectrometry (XRF)
•Inductively coupled plasma (ICP)
•X-ray powder diffraction (XRD)

Experimental Aims

•To determine the usefulness of both qualitative and quantitative XRD analysis as an additional tool for the forensic analysis of duct tapes

- Identify optimum instrumental parameters
- Determine within roll variation
- Determine variation between manufacturers
- Determine variation between batches
- Determine effects of sample handling
  - Liquid nitrogen assisted untangling
  - Stretching from removal of tape from a surface
- Determine sample size limitations

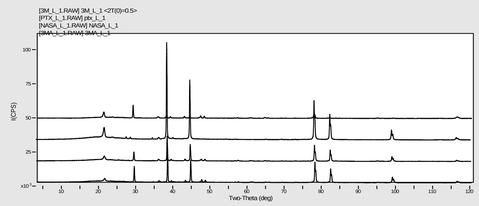
X-Ray Diffraction

•Shortest interatomic distances are 0.5-2.5A, which is consistent with the size of x-rays
•R.I. of x-rays is ~1 for most materials & they are hard to focus w/lenses, so direct visualization is very difficult
•Diffraction requires the wavelength of light to be the same order of magnitude as the repetitive distance between scattering objects
•X-rays are scattered by electron densities periodically distributed in the crystal lattice
•Diffracted waves consist of sharp interference maxima (peaks) with the same symmetry as the electron densities

XRD Advantages

•Non-destructive
•Easy sample preparation
•Does not require a large sample size
•Introduces variables other than elemental composition

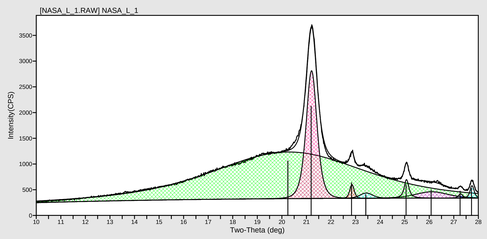
Four different tapes, 2θ 3-120



XRD parameters to be considered

•Data collection specifications
•Data fitting method for polyethylene crystallinity measurement
•Quantitative data used
•Orientation effects

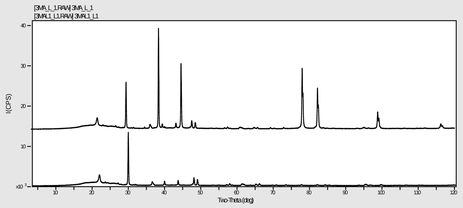
Profile fitting -21.68% Crystallinity



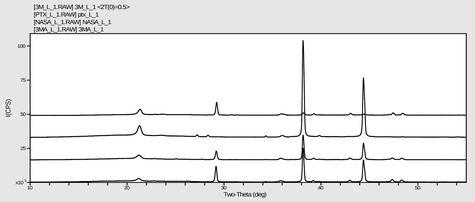
Factors affecting diffractogram peak characteristics

•Peak position	–Unit cell parameters
	–Absorption, porosity
•Peak intensity	–Atomic parameters
	–Preferred orientation,
	–Percent composition/quantity
•Peak shape	–Crystallinity
	–Grain size, strain, stress

Spinning (top) vs Stationary (bottom) sample



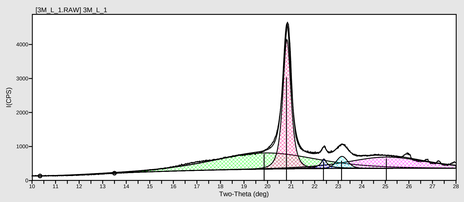
Four different tapes 2θ 10-55



Data Analysis

•Visual inspection of diffractograms
•Jade software
  - Quantitative peak area measurements
  - Percent crystallinity calculations
  - Search/match to library
•Mathematica multivariate statistical analysis

Profile fitting – 57.84 % Crystallinity

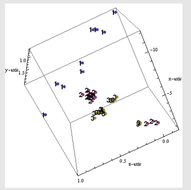


Statistical analysis

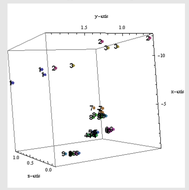
Statistical analysis was started using the Mathematica software. The preliminary trials have involved the diffraction peak areas for 13 peaks that all tape samples have in common.
Once the qualitative differences are factored in, a greater discrimination can be expected.

Principal Component Analysis (PCA)
3M, Nashua & 3M Tough-3D Plot of first 3 eigenvectors

Distinguished by type



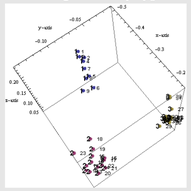
Distinguished by roll



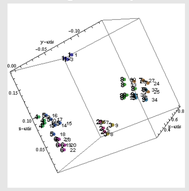
Linear discriminant analysis (LDA) and jackknifing only resulted in two misclassified spectrum in the comparison by type. In the comparison by roll there were 444.

Canonical Variate Analysis (CVA)
3M, Nashua & 3M Tough-3D Plot of first 3 eigenvectors

Distinguished by type



Distinguished by roll



Linear discriminant analysis (LDA) and jackknifing resulted in no misclassified spectrum in the comparison by type and 77 in the comparison by roll.

Conclusions & Continuing work

•So far the method shows some promise in being able to distinguish between duct tapes based mainly on the XRD analysis of the backing
•There is more work to be done in order to determine the viability of method for forensic analysis of duct tapes and the limits of the discrimination that can be achieved

•Continuing efforts will include
  - Obtaining and analyzing more samples
  - Other data fitting equations for the calculation of polymer crystallinity
  - Including more variables in the statistical analysis

Acknowledgements

My thanks to:
-My dissertation mentors, **Dr. Kubic**, **Dr. Reffner** and **Dr. Petraco** for their ongoing help and advice
-**The Academy of Finland** (**Suomen Akatemia**) for their financial support.
-**John Jay College of Criminal Justice** for the use of the instrument and space