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CHARACTERIZATION OF POLYIMIDE VIA FTIR ANALYSIS

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ABSTRACT: Fourier Transform Infrared Spectroscopy (FTIR) can be a useful bulk analysis technique to characterize a wide range of materials, including polyimide. FTIR utilizes the fundamental physics behind spectroscopy and infrared light to obtain a spectrum, representative of the chemical composition of a particular material. Furthermore, this FTIR spectrum represents the material's infrared absorption and transmission creating a unique molecular spectrum. This uniqueness makes FTIR analysis beneficial for evaluating the effects of various conditions on polyimide. For this study, polyimide was subjected to various baking temperatures, chemical processes, and micro-fabrication techniques to determine the influences on FTIR. These influences were then qualitatively correlated to chemical changes within the polyimide's chemical structure. Furthermore, by understanding the effects of such conditions on polyimide, it can become more versatile across various engineering applications.

1. INTRODUCTION

Infrared spectroscopy can be a practical analysis technique in determining molecular and chemical composition of a variety of materials. More specifically, Fourier Transform Infrared Spectroscopy (FTIR) is a useful infrared spectroscopy method that utilizes the fundamental physics behind a spectrometer to obtain a "molecular fingerprint" of a given material.

FTIR analysis can be performed on any partially transparent material to obtain its chemical and molecular composition. FTIR exposes and reflects infrared light onto the given material. From the reflection, a detector obtains the material's absorption and transmission of the infrared light. This data is then translated into a FTIR spectra which is representative of the chemical bonds present within the particular material.

Figure 1: Perkin Elmer Fourier Transform Infrared Spectroscopy (FTIR) instrument.

This study performed a specific application of FTIR analysis to the commercially available polyimide. Polyimide is a commonly used fabrication material due to its flexibility and versatility across various scientific and technological applications.

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To utilize this material in thin film applications, the polyimide has to be subjected to various fabrication processes. These conditions include baking polyimide at a range of temperatures and exposure to commonly used chemicals.

In order to properly use polyimide, it is important to understand both the physical and chemical properties of the material under both typical and modified conditions. Moreover, a thorough scientific understanding and FTIR analysis of polyimide has been performed to better characterize this material under various processing conditions.

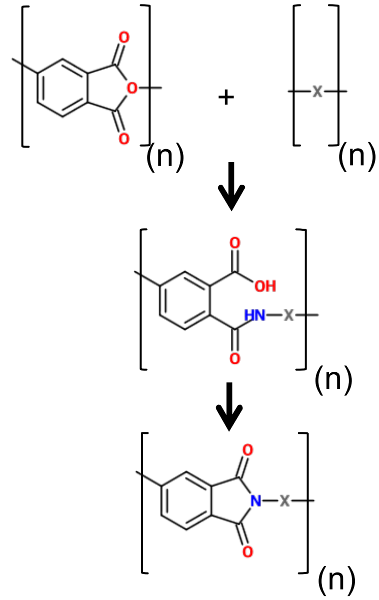

Figure 2: Formation of polyimide from respective polyamic acid precursors.

2. Experimentation

2.1. Preparation

2.1.1. Materials

The polyamic acid precursors used in this study were commercially available polyimide in solvent, N-methyl-2-pyrollidone (NMP). Upon curing at high temperatures process, monomers bonded together to form the full strength polyimide.

2.1.2. Substrates

The substrates used for the characterizing polyimide studies were silicon wafers. Silicon was selected as it is an appropriate substrate material to support the polyimide film through subsequent fabrication processes.

2.1.3. Sample Preparation

All samples were prepared by spinning the polyamic acid precursor of the respective polyimide onto appropriate substrates using a polyimide spinner. The polyamic acid was then soft baked to remove excess NMP solvent from the polyamic acid solution. The samples were then ready for exposure to subsequent fabrication processes.

2.2. Exposure to Fabrication Processes

2.2.1. Motivation

In any micro-fabrication setting, materials must be subjected to a series of fabrication processes. This can range from metal deposition to chlorinated photoresist stripping. Regardless, it is crucial to understand how material will physically and chemically respond to these processes. If materials were to fail or become defective upon exposure to such conditions, then this would be a serious issue for further material usage. Therefore, the motivation behind this study was to determine the capabilities and limitations when using polyimide.

2.2.2. Effects on Polyimide at Various Baking Temperatures

Standard preparation of polyimide is to soft bake the polyamic acid precursor to remove excess solvent. This is followed by a high temperature curing process to imidize polyamic acid to its final polyimide state. Moreover, the FTIR spectra are well known for these two polyimide states, soft bake and cured.

However, the chemical changes that occur during different baking temperatures are unclear. So instead of fully curing the polyimide, a lower temperature-baking step was performed after the soft bake. This baking step was performed with a range of low temperatures to understand how these temperature differences influence chemical changes to the polyimide. Temperature ranges between 100°C and 150°C were studied with respect to polyimide's FTIR spectral changes. .

2.2.3. Effects on Polyimide with Chemical Exposures

When using polyimide in micro-fabrication, the material must also be exposed to a variety of chemicals. Some of these common chemical treatments include acetone, hydrofluoric acid (HF), and silicon etchants. The capability to survive exposure to these chemical is necessary as these chemical are used for various fabrication technique including photoresist stripping, metal etching, and silicon etching. Each chemical was applied to the polyimide in a representative manner to effectively evaluate the treatments' effects.

2.3. FTIR Analysis

The most critical portion of this study was performing an accurate FTIR analysis. In general, the peaks present in a given FTIR spectrum are well understood. Moreover, these peaks can be correlated with a particular chemical bond. Table 1 below relates peak location with corresponding chemical bonds.

Table 1: IR Absorption Table for Relevant POLYIMIDE Peaks

Functional Group	**Absorptions (cm^{-1}) / (Peak Intensity)**
C=O Carboxylic Acid	1760-1690 (s)
C=O Ketone	1715 (s)
C=C Aromatic	1700-1500 (m,m)
C—N Aromatic	1335-1250 (s)

These peaks, relevant to the chemical structure for polyimide, will be evaluated throughout the various fabrication conditions to determine if any significant FTIR changes occur. The changes to be observed will include relative peak heights and formation or deformation of peaks. An additional qualitative comparison will be performed comparing polyimide after exposure to fabrication processes with a reference spectrum of cured polyimide. Also, all spectra used for this analysis are absorption spectra, not transmission spectra.

3. Results & Discussion

3.1. Effects on Polyimide at Various Baking Temperatures

The soft baked polyamic acid precursor of polyimide was baked at various temperatures, between 100-150°C, instead of fully curing at high temperatures. The influence on the FTIR spectra at these temperatures was then evaluated.

It was evident from this spectral analysis that baking at this lower temperature range provided significantly different FTIR spectra than that of cured polyimide. More specifically, peaks at ~1700cm^{-1} and ~1350cm^{-1} were noticeably different. Upon detailed analysis the FTIR differences were consistent with the expected chemical changes.

The peak at ~1700cm^{-1} most likely corresponds to a C=O stretch for ketone functional group. In fully cured polyimide this bond is more abundant than it is lower temperature baked counterpart. This chemical understanding indicates as to why the FTIR is significantly different at this location.

The peak at ~1350cm^{-1} most likely corresponds to a C—N stretch functional group. Once again, in fully cured polyimide, the C—N stretch is more abundant. Therefore, the peak is expected and experimental observed as being significantly more intense.

Moreover, the FTIR changes observed in this analysis are indicative that polyimide baked at lower temperatures instead of curing at higher temperatures has significantly different chemical properties. Furthermore, these chemical properties will possibly alter the polyimide when it comes to other fabrication processes.

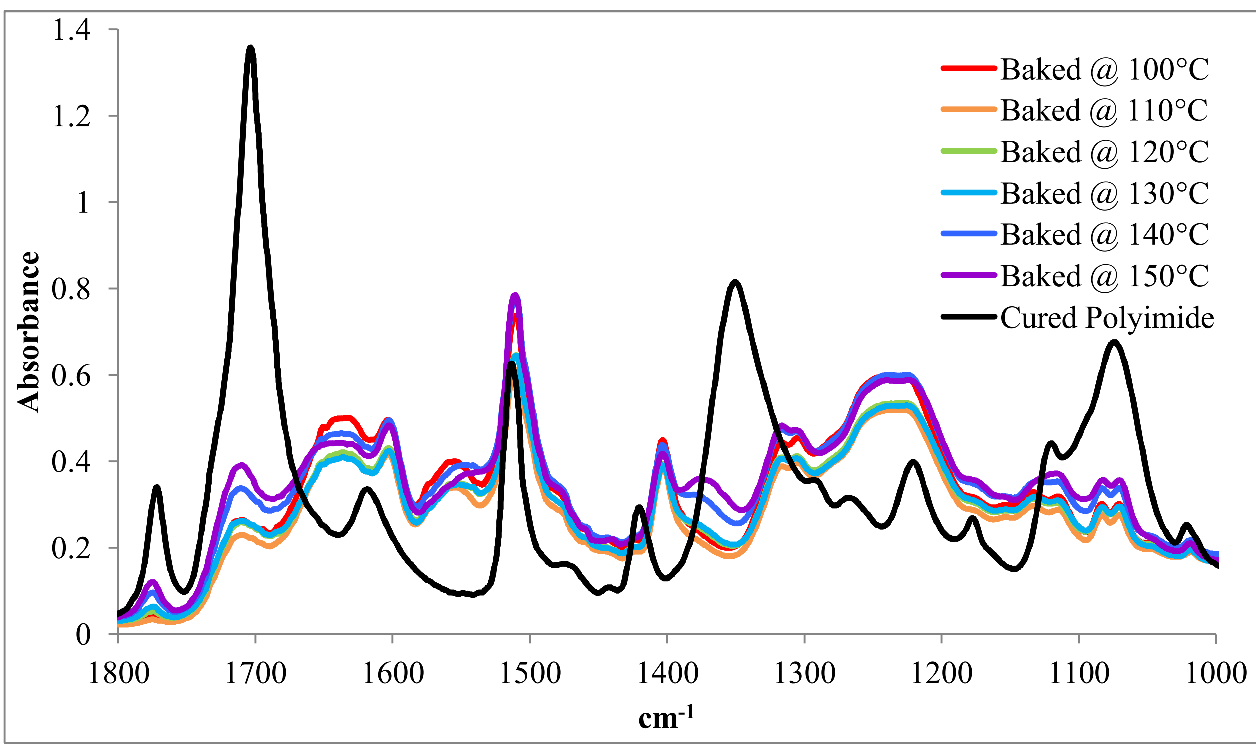

Figure 3: FTIR spectra of baking at various temperatures. These are compared to a reference spectrum of fully cured polyimide.

3.2. Effects on Polyimide with Chemical Exposure

When it was determined that polyimide baked at lower temperatures had significantly different chemical properties, a concern arose with the durability of low baking temperature polyimide when exposed to several chemicals. Therefore, polyimide baked at 120°C was exposed to acetone and HF. These FTIR spectra obtained after these treatments were then compared to the FTIR spectrum of baking at 120°C without subsequent chemical exposure.

It was evident that acetone soaking provided very little FTIR changes compared to similar polyimide without the chemical exposure. Moreover, it was concluded that acetone soak did not significantly alter the polyimide chemistry.

With the hydrofluoric acid (HF) exposure, there were no drastic FTIR differences. In general, the HF exposure had an intensity bias in which all the peak were larger.

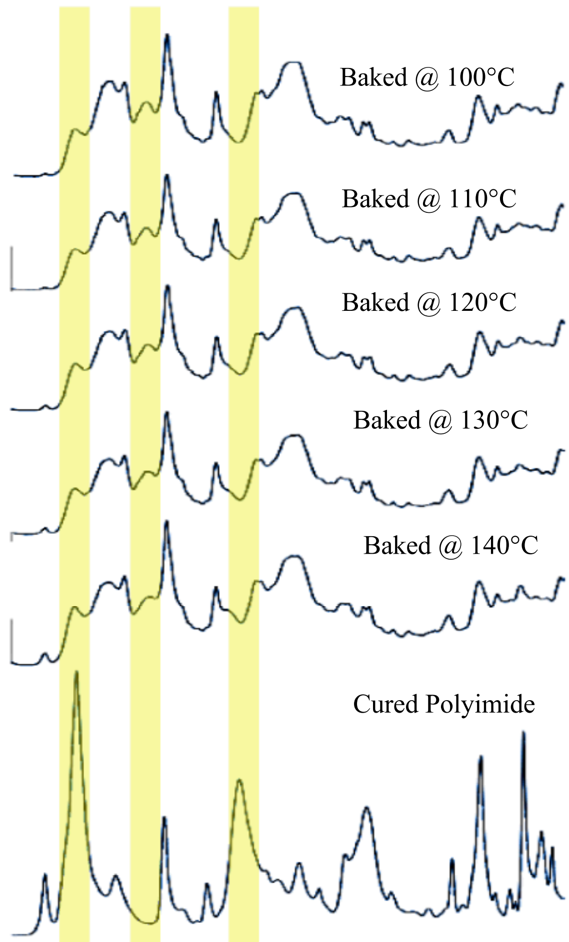

Figure 4: Progression of FTIR spectral changes with increasing baking temperature.

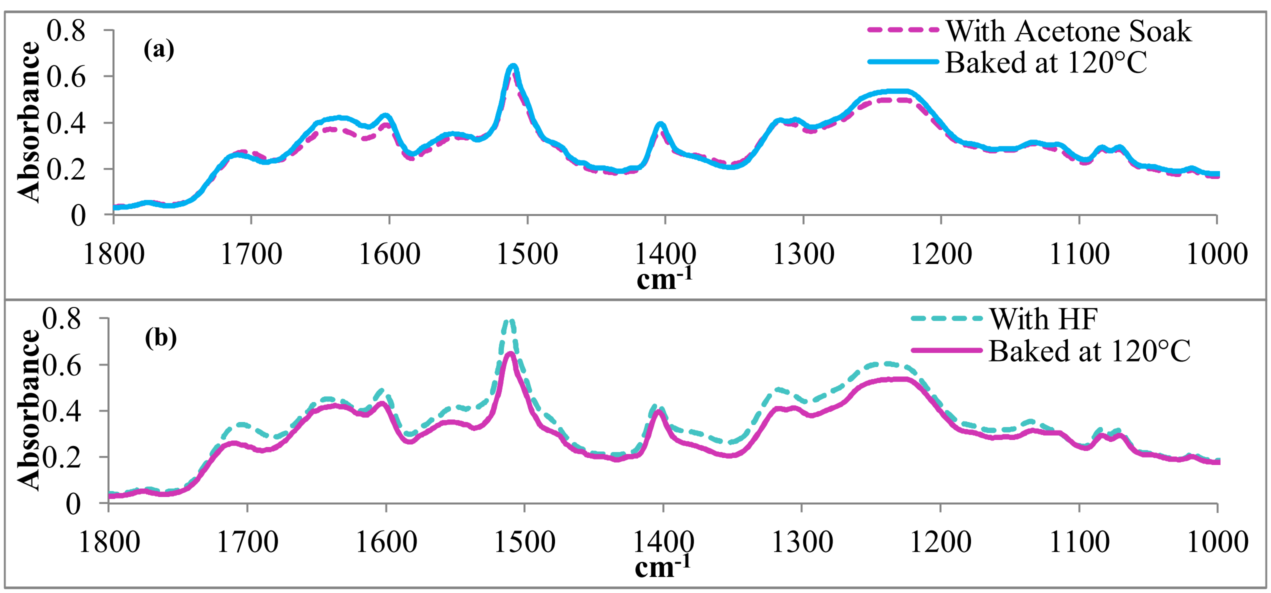

Figure 5: (a) FTIR spectral comparison after acetone soak. (b) FTIR spectral comparison after HF.

However, there were no significant peak differences between the chemically exposed and non-exposed. Therefore, it was concluded that when using a polyimide with a significantly chemical composition than fully cured polyimide, the polyimide can survive various chemical exposures.

4. Conclusion

In conclusion, an in-depth FTIR analysis was performed to better understand and characterize polyimide. Moreover, by FTIR spectral changes to the polyimide were observed after exposure to various micro-fabrication processes. The processes included varying baking temperature and several chemical exposures. Furthermore, FTIR spectral changes were associated with polyimide's chemical changes under such fabrication conditions.

By performing this detailed evaluation of polyimide changes during very representative processes, a better understanding of the polyimide's versatility and usability in micro-fabrication was obtained. Also, the knowledge gathered in this study may be beneficial for a range of different scientific, technological, and engineering applications.

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