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Thermal Analysis of Bismaleimide/Graphite Fiber Composite by TG/FTIR

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1993

THERMAL ANALYSIS OF BISMALEIMIDE/GRAPHITE FIBER COMPOSITE BY TG/FTIR

A Thesis
Presented to
the Faculty of the Department of Chemistry
Western Kentucky University
Bowling Green, Kentucky

In Partial Fulfillment
of the Requirements for the Degree
Master of Science

by
Qi Zhang
August 1993

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THERMAL ANALYSIS OF BISMALEIMIDE/GRAPHITE FIBER COMPOSITE BY TG/FTIR

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THERMAL ANALYSIS OF BISMALEIMIDE/GRAPHITE FIBER COMPOSITE BY TG/FTIR

Name Qi Zhang Date May, 1993 46 Pages

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The purpose of this project was to study the thermal and oxidative stability of a specific polymer composite. A TG/FTIR combined technique was employed to characterize this polymer/graphite fiber composite. The polymer composite sample was decomposed in nitrogen, air, and 3% oxygen atmospheres in a Seiko thermogravimetric analyzer. The use of a gas cell Fourier-transform IR spectrometer downstream from the TGA allowed for the identification of evolved gas products.

The thermal oxidative stability (TOS) of this composite was compared to its thermal stability. Also, comparison of the TOS in different atmospheres was made. In addition, the effects of specimen geometry in terms of sample thickness and surface area-to-volume ratio (A/V) were investigated by relating gas diffusion into the specimen to its relative stability.

ACKNOWLEDGEMENTS

With deep gratitude I wish to express my sincere appreciation to Dr. Wei-Ping Pan, whose interest, attitude and aptitude have been of invaluable service to me during my period of study under his guidance. I would also like to extend my deepest appreciation to Dr. W. M. Lee for his guidance.

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INTRODUCTION

The thermal oxidative stability (TOS) of advanced polymer composites is an important material selection consideration for commercial aircraft applications. In this project, the thermal oxidative stability and thermal stability of a bismaleimide/graphite fiber composite, X5260/IM7, were studied by the use of dynamic thermogravimetry/gas cell Fourier transform infrared spectrometry (FTIR) analysis.[1]

Thermal analysis (TA) techniques have been used in polymer studies for research, development and in quality control, as well as for analytical and comparison purposes for many years.[2] In the thermal analysis technique of thermogravimetry (TG) the mass of a substance is continuously monitored as a function of temperature and/or time. Three modes of thermogravimetry are commonly used: (a) isothermal thermogravimetry, in which the sample mass is recorded as a function of time at constant temperature; (b) quasi-isothermal thermogravimetry, in which the sample is heated to constant mass at each of a series of increasing temperatures; and (c) dynamic thermogravimetry, in which the sample is heated in an environment where temperature changes in a predetermined manner, preferably at a linear rate.[3]

Dynamic thermogavimetry is the most widely used simple and rapid method. Sometimes, the results can be extrapolated to obtain the life expectancy of a composite at lower temperatures. It has been found that the TG curve obtained at a heating rate of 150°C/hr corresponds roughly to aging data obtained from isothermal aging over 10-60 min period,[4] which means that the weight loss after reaching a certain temperature in the thermobalance would be the same as if the material had been exposed to this temperature over a period of 10-60 min.

The thermal analysis technique does not, however, identify the gas evolved; that information is essential for complex decompositions. The use of Fourier transform infrared spectrometry (FTIR) for evolved gas analysis has been reviewed by Lephardt.[5] The potential use of FTIR for the identification of products of thermal degradation were suggested by Liebman et al.[6] The advantages and capabilities of interfacing a thermogravimetric analyzer and Fourier transform IR spectrometer for studying polymeric materials have also been reported.[7,8] Among the various physical methods used for the analysis of thermal degradation products, infrared spectroscopy has often been found very powerful because it is versatile, rapid, and accessible.[9]

There are two general types of commercial polyimides: thermoplastic and cross-linked. Thermoplastic polyimides (condensation-type) are derived from a condensation reaction between anhydrides or anhydride derivatives and diamines, whereas cross-linked polyimides (addition-type) are derived from an addition reaction between unsaturated groups of the preformed imide monomers or oligomers.[10]

In recent years, the class of addition-type polyimides has been investigated extensively. Bismaleimides are of particular interest[11] because of their easy availability, low price, excellent processing characteristics, and outstanding thermomechanical and flammability behavior at elevated temperatures in the final cured state.

The basic structures of bismaleimides are shown in Figure 1. The bridge between the maleimide or succinimide rings can be either aliphatic bridges or aromatic bridges. Stenzenberger et al.[11] reported that the polybismaleimides with aromatic bridges between the maleimide residues are thermally more stable than those with aliphatic polymethylene sequences, and the aliphatic bridges are thermally less stable than the succinimide rings.

Bismaleimides can be converted to homopolymers, copolymers or terpolymers and used as cross-linking agents. The first successful homopolymerization of bismaleimides into thermoset polymers was accomplished by F. Grundschober and J. Sambeth in 1968.[12] Practical application of bismaleimides to polymeric systems required copolymerization with amines or nucleophylic monomers by means of the Michael addition reaction, or with olefinic or acetylenic monomers, as illustrated in Figure 2.[10]

H. D. Stenzenberger et al.[11] investigated the thermal decomposition of polybismaleimides by using programmed and

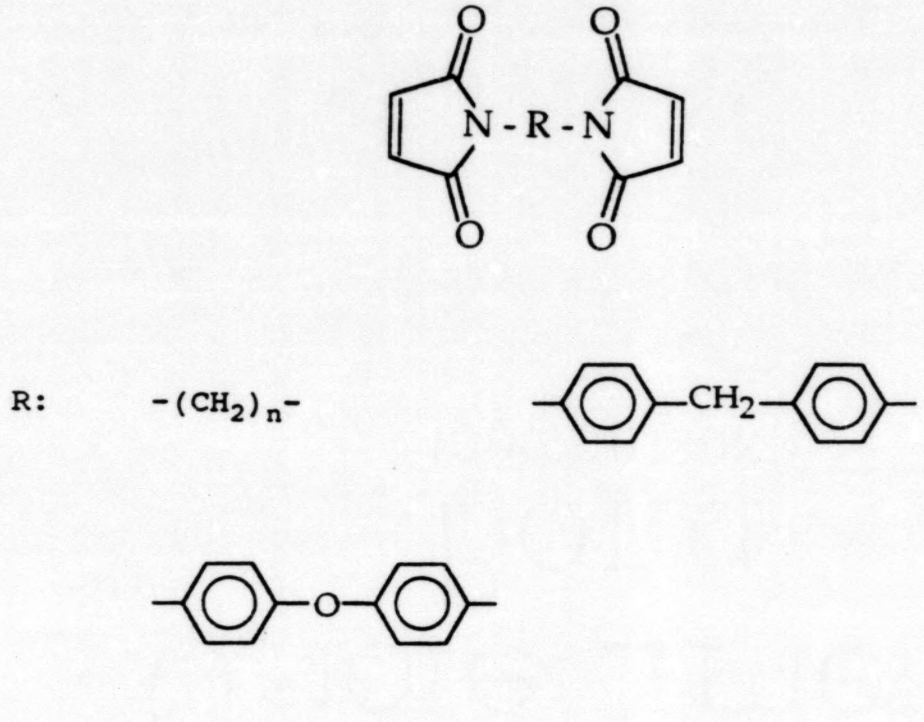

Figure 1. Basic structure of bismaleimides.

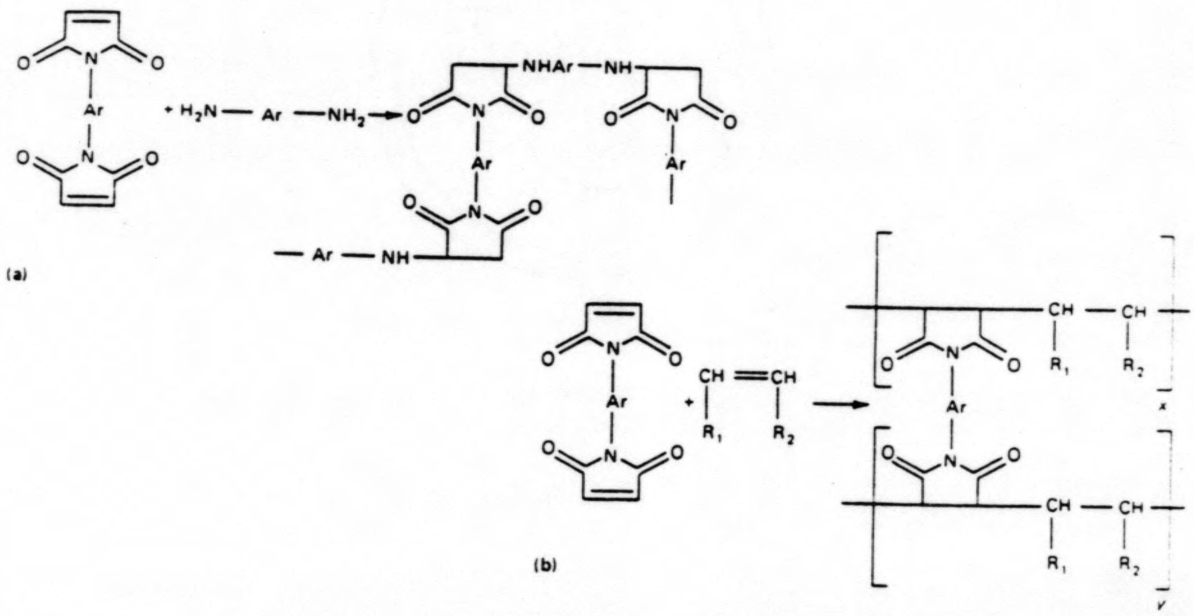

Figure 2. Bismaleimide addition-type polyimides. (a)Michael addtion BMIs. (b) Bismaleimide-olefin copolymers.

isothermal thermogravimetric analysis (TGA) in nitrogen. In addition to the TGA, the pyro-field ion mass spectra(PFIMS) of the polymers were recorded and discussed.

BASF reported processing capability, exceptional mechanical properties, enhanced toughness and improved thermo-oxidative stability on their newly developed commercialized bismaleimide (BMI), Rigidite* X5260 resin system.[13] Also, with its excellent properties of toughness and hot/wet performance, X5260 can be considered as a prime candidate for primary aircraft composite structures.

Graphite fibers have been made from a number of precursors, but are now mainly produced from polyacrylonitrile (PAN), pitch and rayon.[14] An outstanding feature of graphite fiber is its resistance to fatigue and corrosion.

Graphite fibers are inert to temperature and humidity over the range of most resin service temperatures. Their combination of strength, stiffness, fatigue resistance, density, and toughness is unique in the advanced-composite field.

PAN-based carbon fibers represent, by far, the most important carbon fibers used today. Hercules graphite fibers are produced in a continuous operation in which the polyacrylonitrile precursor undergoes a series of precisely controlled processes.[15] Exposure to extremely high temperatures chemically changes the precursor, yielding high strength-to-weight and high stiffness-to-weight properties through oxidation, carbonization and graphitization. The successive surface treatment and sizing stages improve bonding and ease in handling.

Hercules Type IM7, an intermediate modulus and high tensile strength PAN-based carbon fiber, is the continuous reinforcing fiber used in this polymer composite X5260/IM7.

In this study, the thermal oxidative stability (TOS) of X5260/IM7 was determined in terms of weight loss characteristics and evolved gas products. The difference of TOS in air and in 3% oxygen balanced in nitrogen atmosphere was studied. The sample size effect on TOS was also examined and discussed.

EXPERIMENTAL

Bismaleimide/graphite fiber composite samples were obtained from the Dow Chemical Company. There are two forms of test samples, one is the powder form, and the other is the laminated fiber form cut from laminates having varying thickness and surface area-to-volume ratios (A/V). Laminate composite samples were employed for sample size effect assessment. Table 1 lists the various sample dimensions used in this study.

Test samples were pre-dried for about three days at 93.3°C to evaporate moisture and weighed daily until constant weight was attained.

Used in this study was a Seiko TG/DTA 200 Simultaneous Thermo-Gravimetric/Differential Thermal Analyzer connected to a SSC 5040 TA Disk Station. This system followed the composite weight change at a heating rate of 10°C/min with a gas flow rate of 150 mL/min for air and 3% oxygen, 100 mL/min for nitrogen. The TGA was used to characterize the thermal oxidative stability and thermal stability of the composite, and to compare the TOS in different atmospheres.

In order to study the evolved gas products during the composite degradation, a modified Perkin Elmer 1650 Fourier

Table 1. The sizes, dimensions and surface area-to-volume ratio for six samples

Sample No.	Sample Size	Dimensions (LxWxT)	A/V
1	20 mg	0.15"x0.15"x0.042"	74
2	50 mg	0.15"x0.15"x0.1"	47
3	80 mg	0.15"x0.15"x0.172"	38
4	0.6 g	1.0"x0.5"x0.042"	54
5	1.3 g	1.0"x0.5"x0.1"	26
6	2.1 g	1.0"x0.5"x0.172"	18

Transform Infrared Spectrophotometer (FTIR) with gas cell was coupled to a DuPont 951 Thermogravimetric Analyzer with a TA Instruments 2000 Controller. The components were coupled by a connecting heated gas line (an insulated teflon tube, with a temperature of about 200°C) to monitor the evolved gases.

Figure 3 shows the schematic diagram of the instrument used in the TG/FTIR study. A Barnant thermocouple controller maintains the gas cell temperature at about 200°C to prevent condensation of the evolved gas products. The detail of the gas cell, the most important feature in this method, is shown in Figure 4.

The coupled TG system was heated at 10°C/min from 20°C to 900°C under a 50 mL/min air or nitrogen atmosphere to ensure a good sampling of the evolved gases. To control the FTIR sampling, Galactic Software's Labcalc was employed to do the automatic sampling (100°C to 900°C) at a rate of eighty spectra per run.

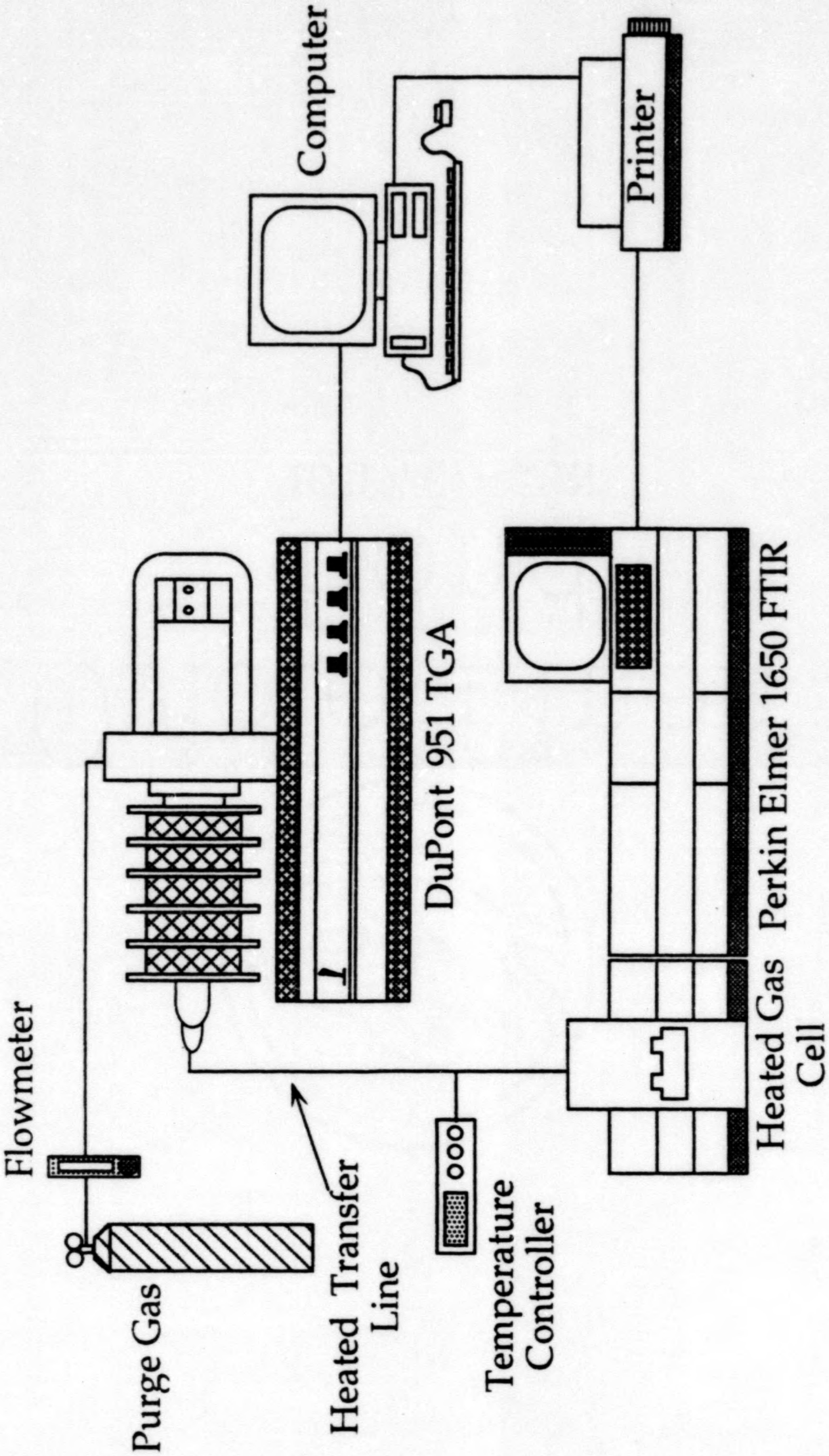

Figure 3. Schematic diagram of the TG/FTIR instrumentation.

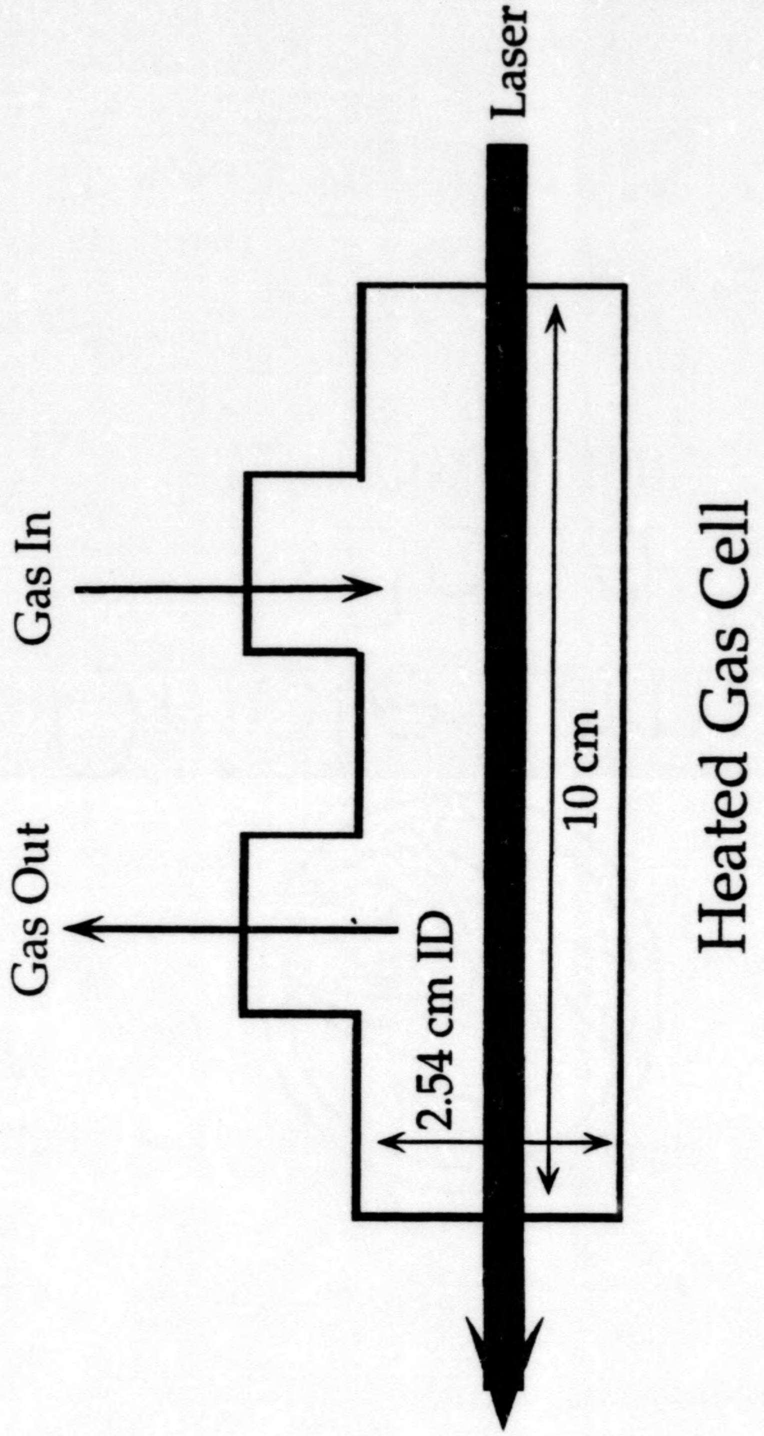

Figure 4. Diagram of the heated gas cell for IR analysis.

RESULTS AND DISCUSSION

I. TGA results

A. Thermal oxidative stability in different atmospheres.

At 10°C/min heating in air, the composite began to lose weight at about 352°C, regardless of its specific thickness or surface area-to-volume ratio. Figures 5, 6 and 7 show the thermogravimetric analysis (TGA) curves of three samples (sample numbers, 1-3) together with the differential thermogravimetry curves (DTG) that were recorded simultaneously. Their weight loss typically displayed three maximum weight change rates with respect to temperature. For example, these three maxima occurred at 421°C, 582°C (as a shoulder) and 657°C (Figure 5) for sample 1. The corresponding weight losses are 5%, 24%, and 54%, respectively. The first peak corresponding to a 5% weight loss is mainly due to volatilized components in the polymer. The second peak corresponding to a 24% weight loss is due to polymer oxidative degradation products. The third peak corresponding to about 54% weight loss is associated not only with the continuing oxidation of any polymer residue but also more than likely with carbon fiber degradation.

Tables 2 and 3 summarize the TG/DTG results of the composite in an air atmosphere for samples 1 - 3.

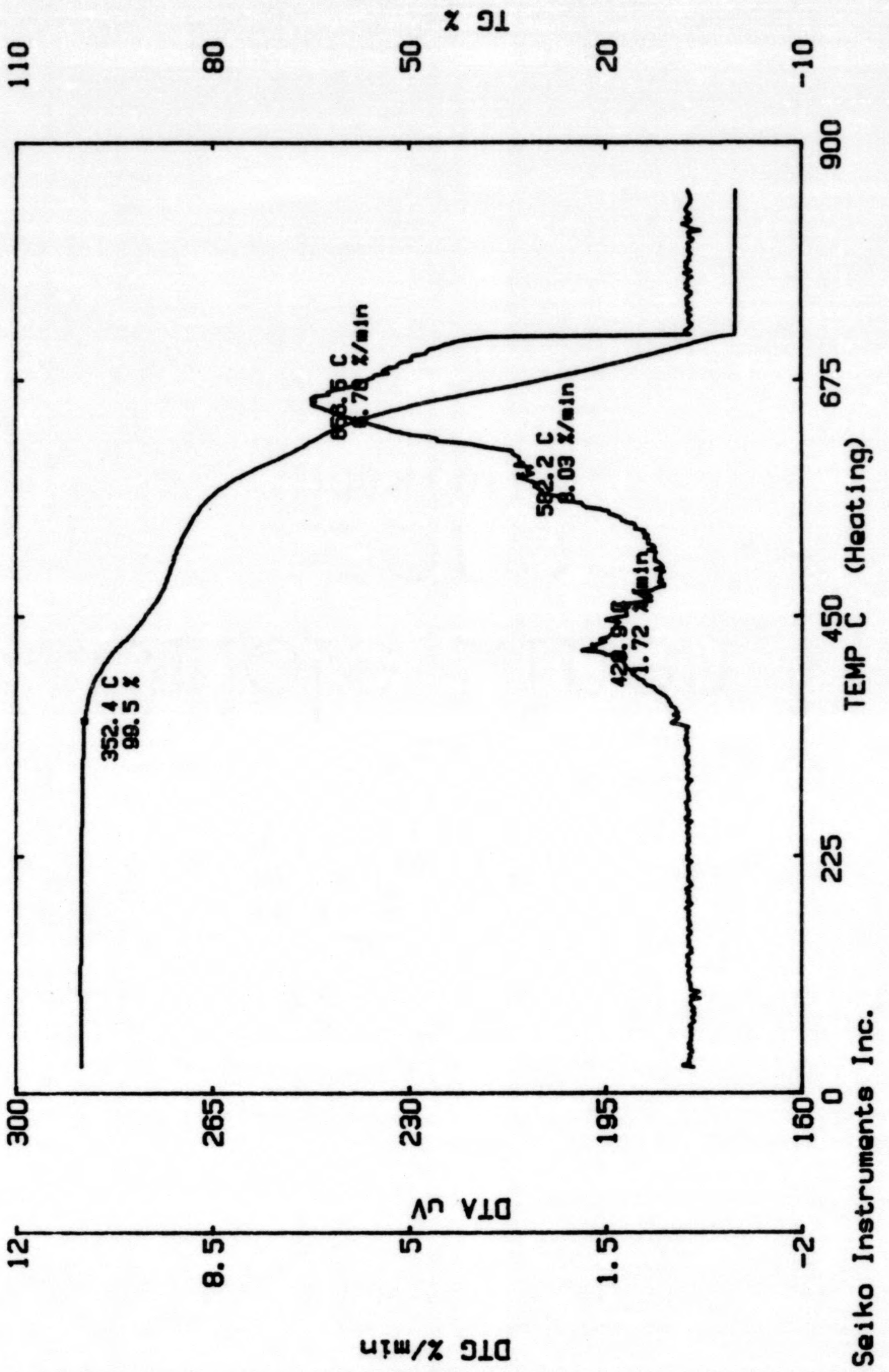

Figure 5. TG/DTG curve of sample 1 in air.

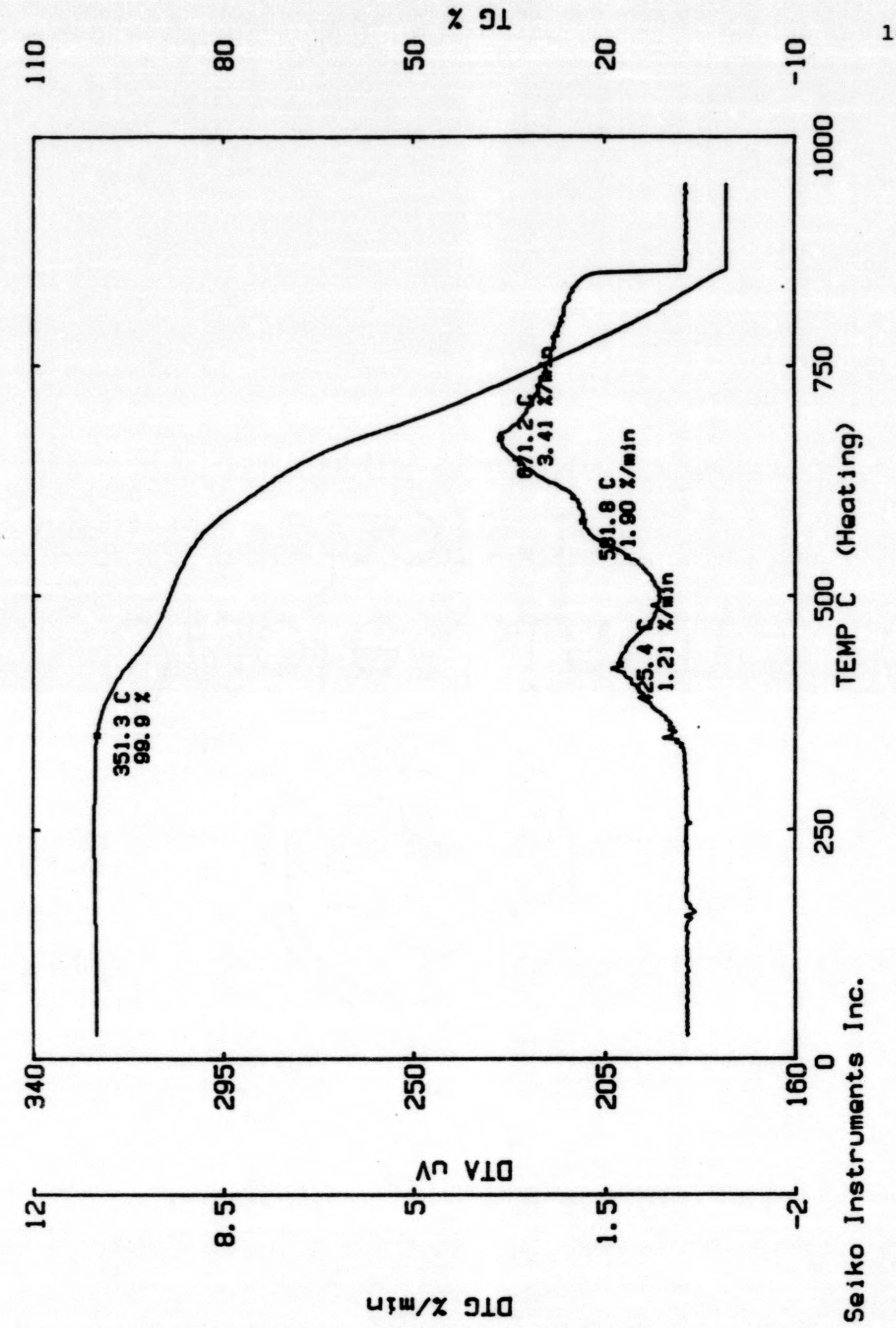

Figure 6. TG/DTG curve of sample 2 in air.

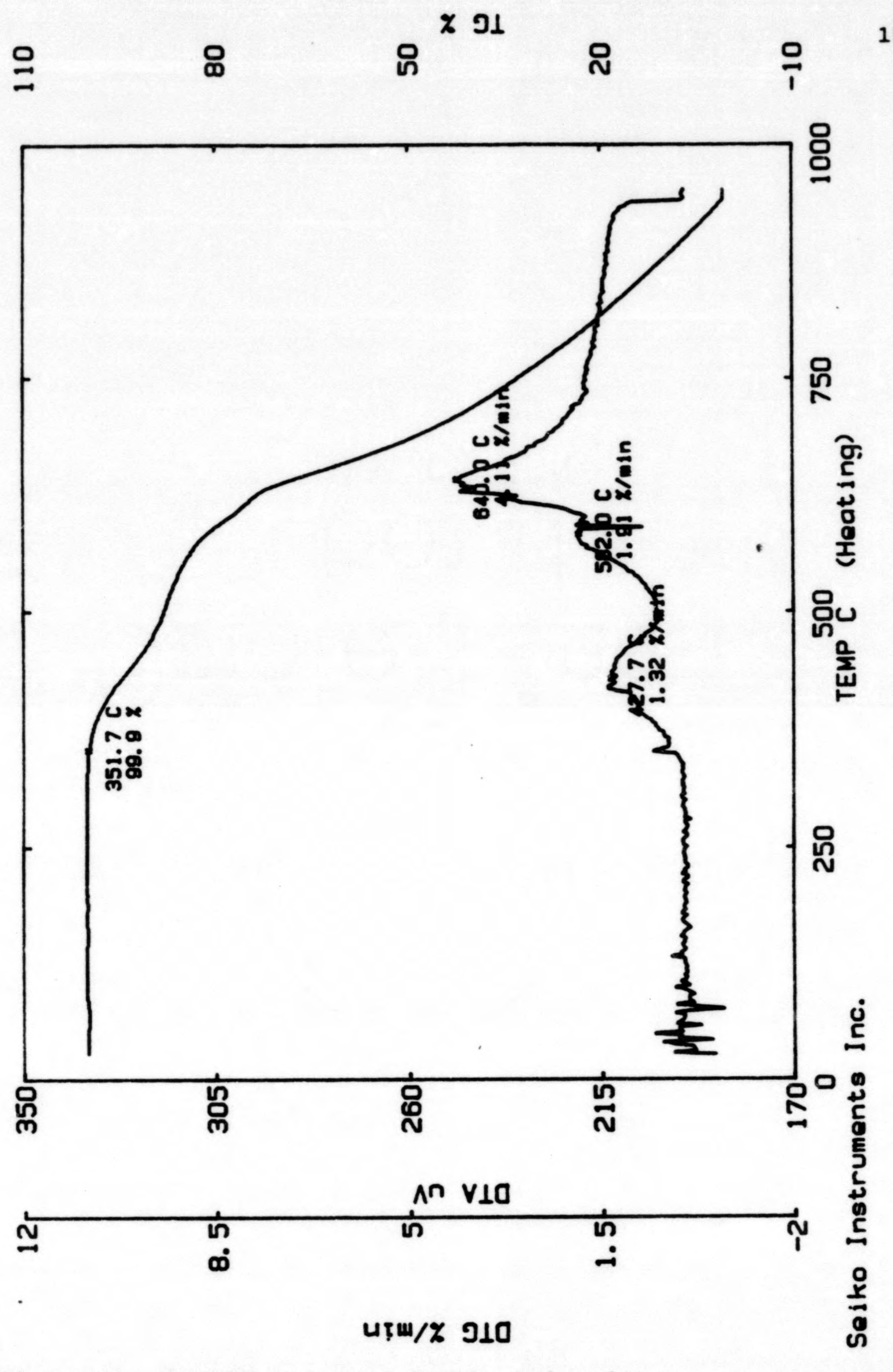

Figure 7. TG/DTG curve of sample 3 in air.

Table 2. Summary of the temperatures (Ti, Tmax, Te) for samples 1-3 heated in air

Sample No.	T_i	T_{max1}	T_{max2}	T_{max3}	T_e
1	352°C	421°C	582°C	657°C	721°C
2	351°C	423°C	582°C	674°C	857°C
3	352°C	428°C	582°C	640°C	938°C

Parameters: T_i is the onset temperature of the initial degradation; T_{max1} is the temperature of the first maximum weight loss rate; T_{max2} is the temperature of the second maximum weight loss rate; T_{max3} is the temperature of the third maximum weight loss rate; T_e is the end test temperature.

Table 3. Summary of the percentage weight losses for samples 1-3 during combustion

Sample No.	W_1 (%)	W_2 (%)	W_3 (%)	W_t (%)
1	5	24	54	100
2	5	18	42	99.3
3	5	18	31	98.8

Parameters: W_1 is the percentage weight loss at T_{max1}; W_2 is the percentage weight loss at T_{max2}; W_3 is the percentage weight loss at T_{max3}; W_t is the total percentage weight loss at T_e.

The temperatures at 5% and 10% weight losses were determined for samples 1 - 6 and given in Table 4. The temperature for 5% weight loss increased slightly with increasing sample thickness above 0.042" and decreasing sample surface area to volume ratio below 74. The temperature at 10% weight loss followed a similar trend. The sample size and thickness effects shown in Figure 8 are indicative of a gas diffusion controlled oxidation and degradation process.

The weight loss in test specimens No. 4 - 6 was measured in the following manner. The sample was put in a quartz tube that was extended out into the Seiko furnace. The sample was heated untill 5% weight loss could be observed initially. After doing so, it was reheated untill 10% weight loss was obtained, and both temperatures were recorded. At the end of the TGA run (1000°C), total weight losses of samples 4 - 6 amounted to 100%, 64% and 51%, respectively. Thus, the thickest and smallest A/V ratio sample (No. 6) produced the least weight loss. Again this behavior suggests a gas diffusion controlled mechanism.

The TG/DTG curve for the powder form of the composite heated in an air atmosphere, is shown in Figure 9. This sample began to lose weight at 309°C, or more than 40°C earlier than that of the laminated fiber form. Its three temperatures at the maximum weight loss rates were all lower than the laminated samples, occurring at 384°C, 532°C, and 639°C.

Table 4. Temperatures at 5% and 10% weight losses for six samples heated in air

Sample No.	A/V	Thickness,in.	T at 5%Loss	T at 10%Loss
1	74	0.042	421°C	457°C
2	47	0.100	425°C	470°C
3	38	0.172	427°C	473°C
4	54	0.042	424°C	468°C
5	26	0.100	445°C	500°C
6	18	0.172	470°C	520°C

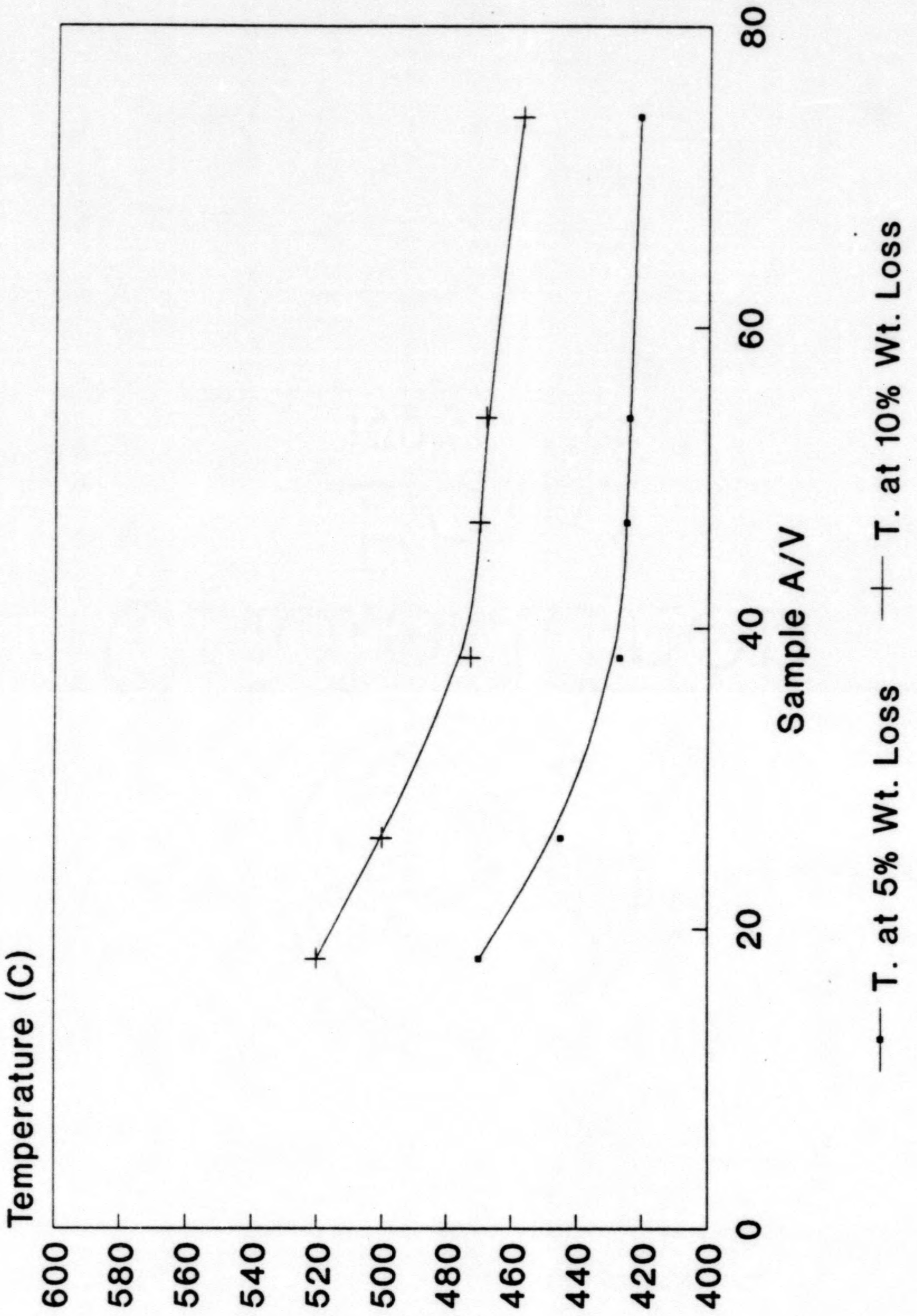

Figure 8. Temperature vs. sample surface area-to-volume ratio.

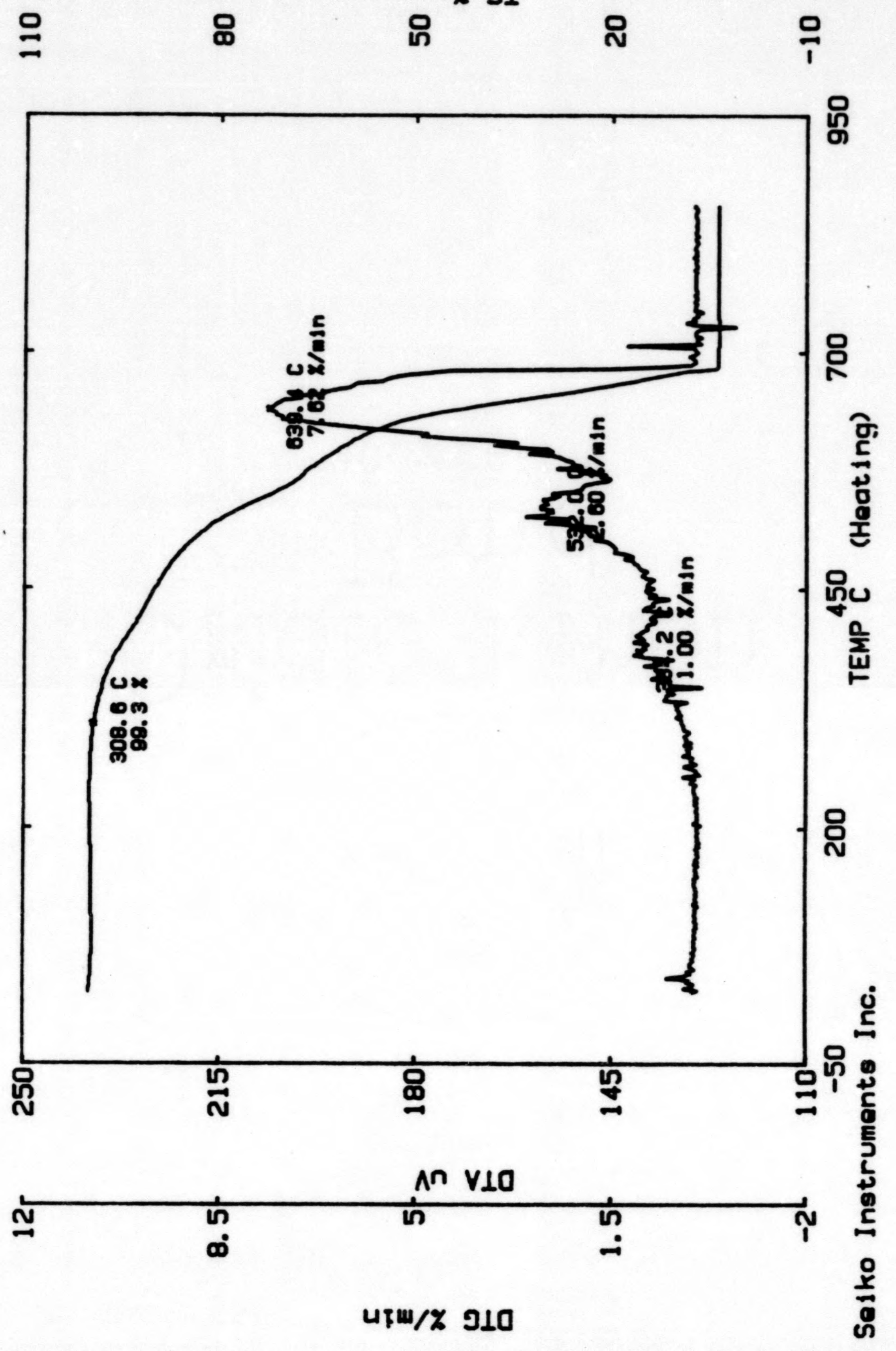

Figure 9. TG/DTG curve of powder sample in air.

At 10°C/min heating, the composite in a 3% oxygen atmosphere also began to lose weight at approximately 352°C. Figures 10 - 12 show the thermograms of samples 1 - 3 in 3% oxygen atmosphere. Even though the initial weight loss temperature for the composite heated in 3% O_2 is the same as that in air, its weight loss rate is relatively small. In the end, the composite lost only 50% of its weight (Figure 11) after heating in 3% O_2. Hence, this TG experiment confirmed the greater composite thermal oxidation stability in a reduced oxygen atmosphere, as expected (Figure 13).

The powder form of the composite began to lose weight in 3% oxygen atmosphere at about 309°C, as shown in Figure 14. A comparison of the thermogravimetric analysis results for the powder form and the laminated fiber form indicates that the latter is approximately 40°C more stable than the former one.

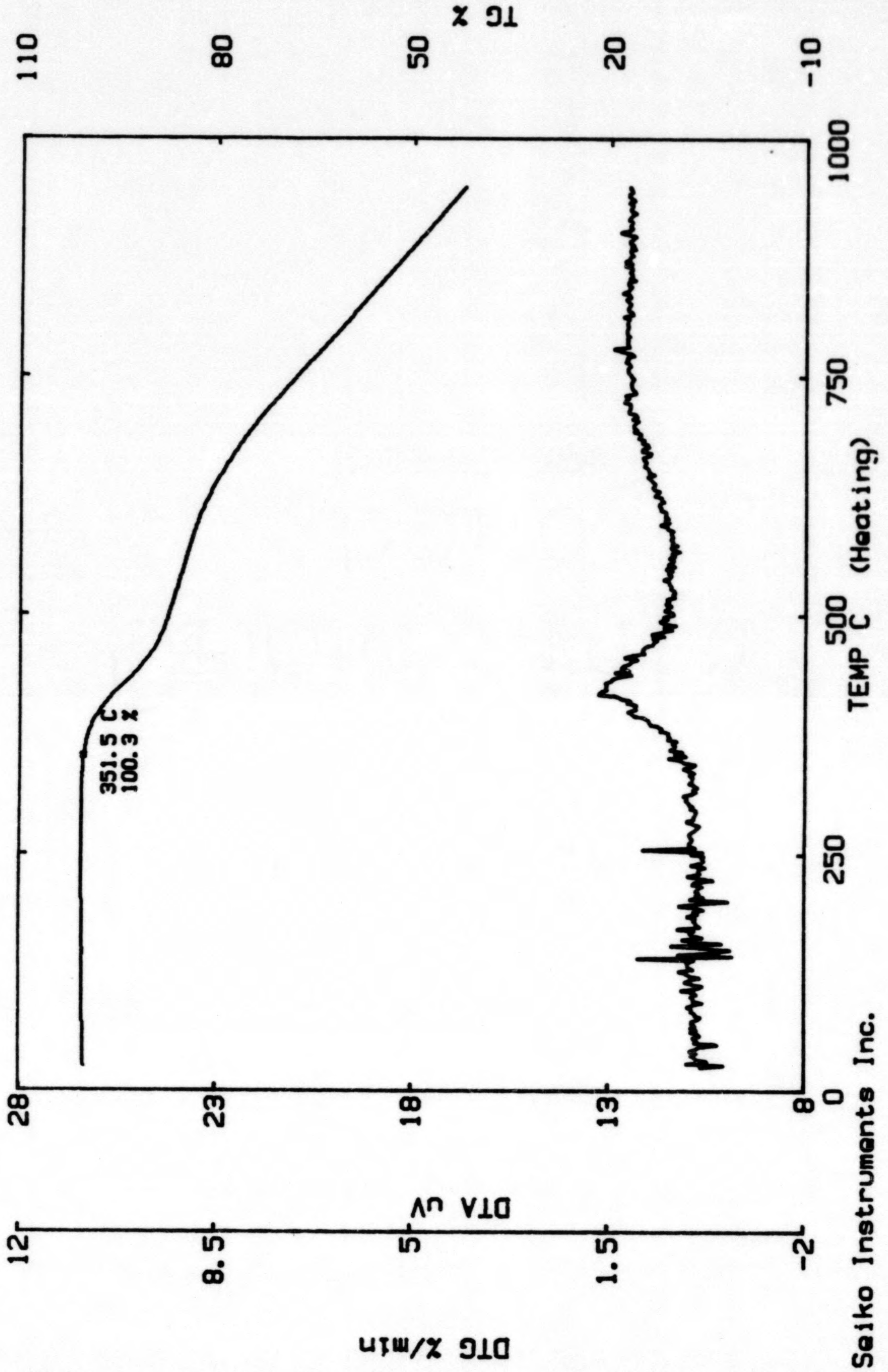

Figure 10. TG/DTG curve of sample 1 in 3% oxygen.

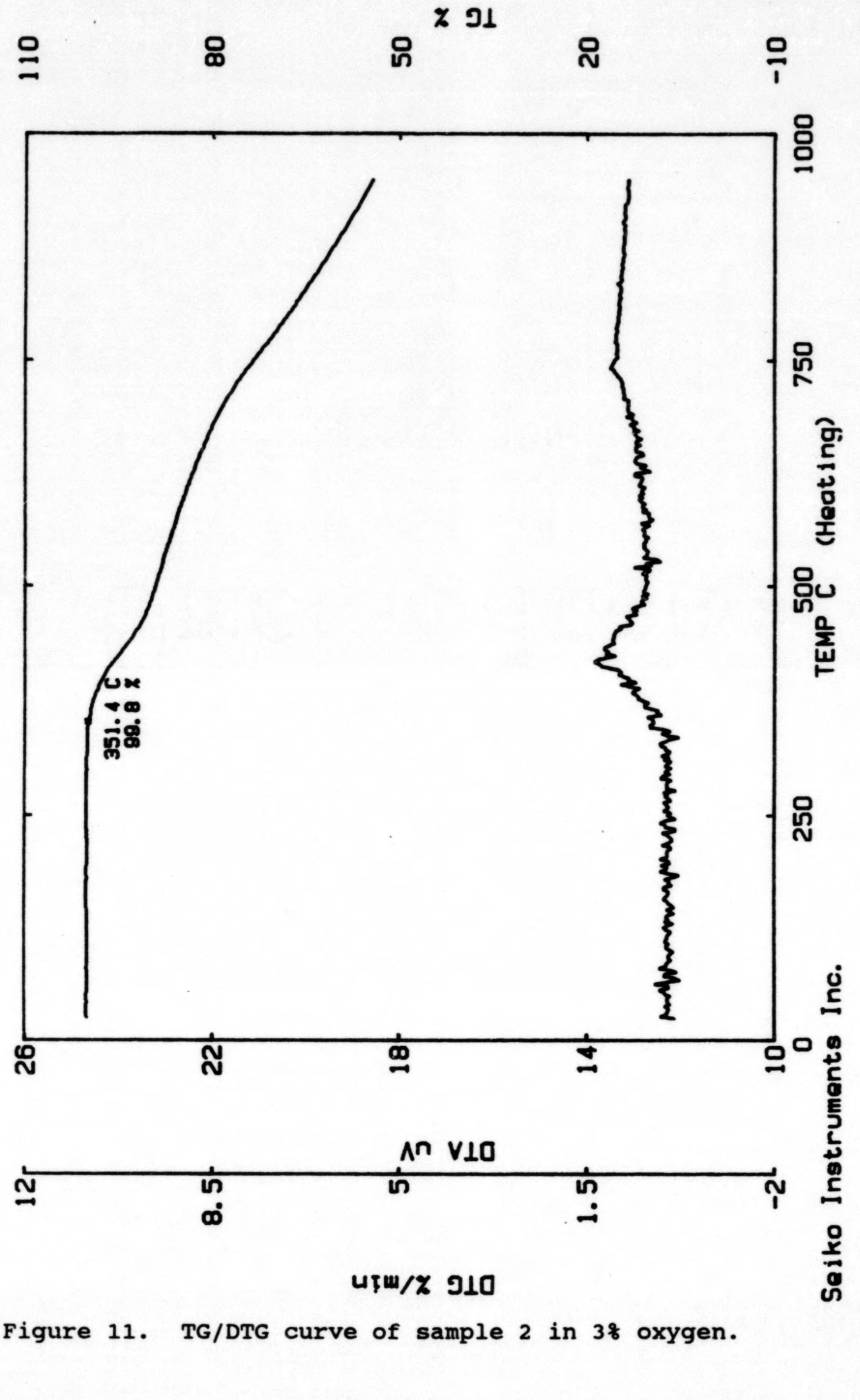

Figure 11. TG/DTG curve of sample 2 in 3% oxygen.

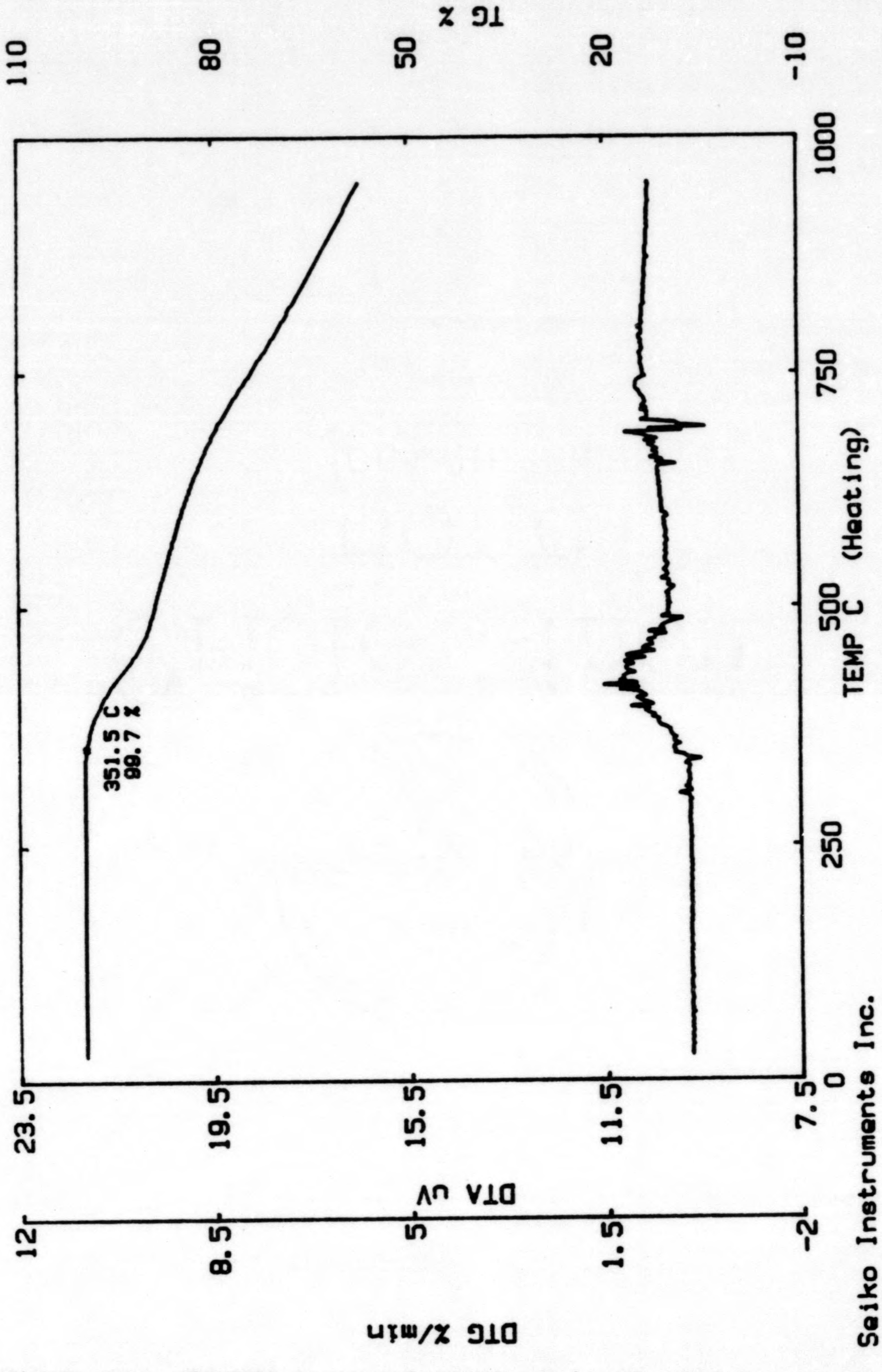

Figure 12. TG/DTG curve of sample 3 in 3% oxygen.

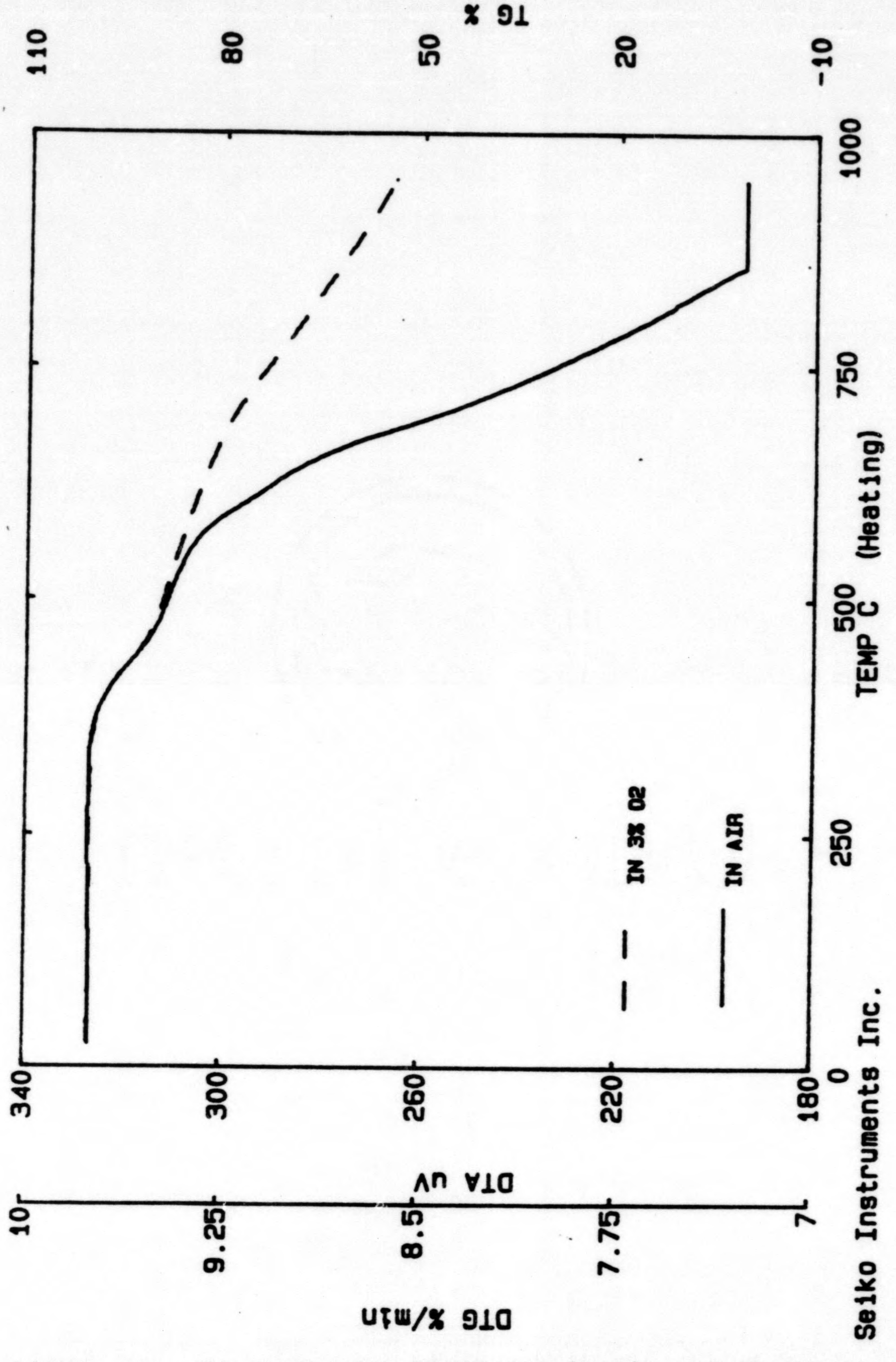

Figure 13. TG curves for X5260/IM7 in air and 3% oxygen.

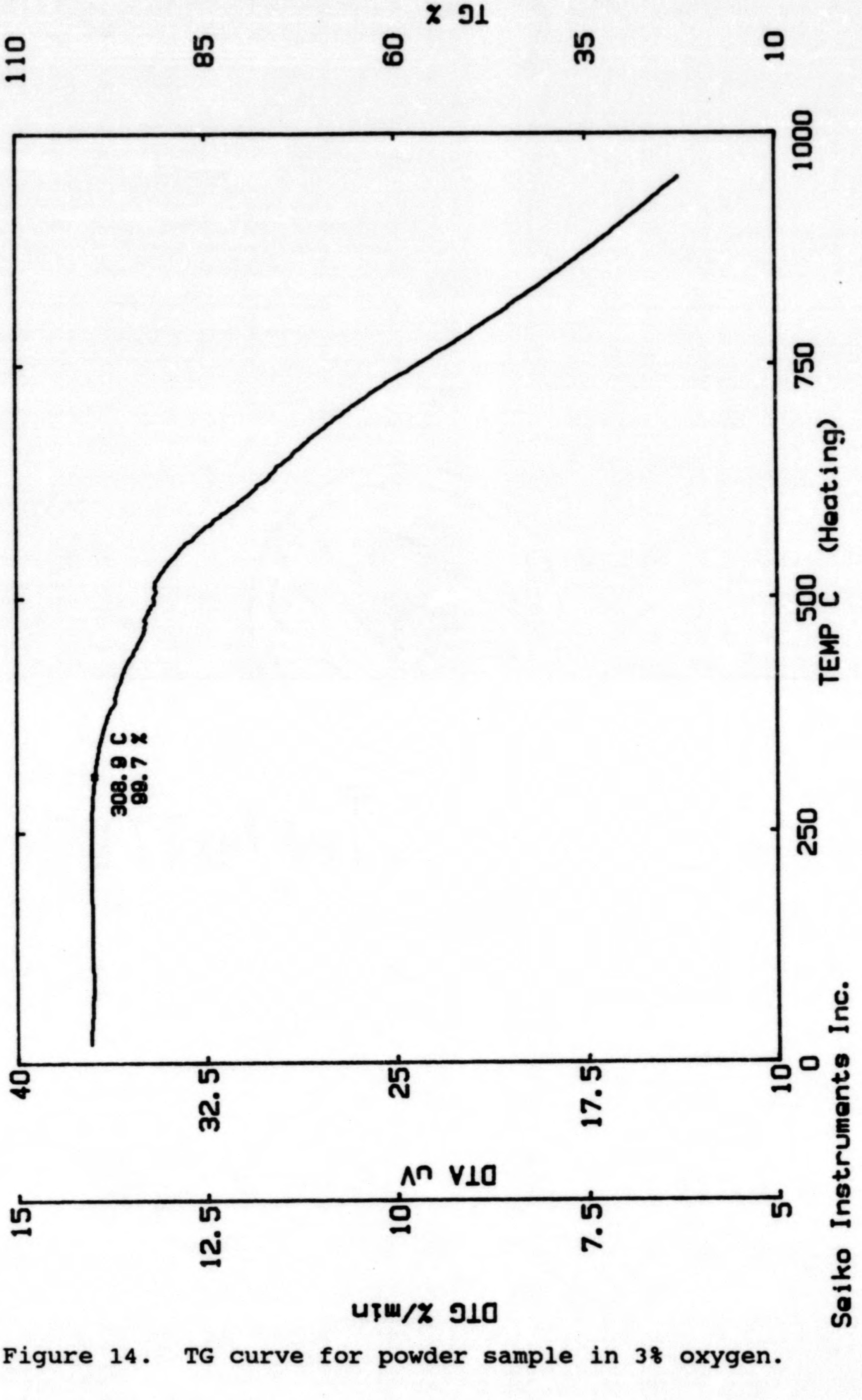

Figure 14. TG curve for powder sample in 3% oxygen.

B. Thermal stability

In a nitrogen atmosphere, the composite began to lose weight at about 345°C. Figures 15 - 17 present the TGA and DTG curves of 3 samples (sample 1 - 3) investigated during pyrolysis. Two T_{max} temperatures at 421°C and 554°C are noted for sample 1 (Figure 15). The corresponding weight losses for the two temperature ranges are 5% and 15%, respectively. The TG/DTG results obtained for samples 1 - 3 are given in Table 5.

The overall weight losses in sample No. 4 through sample No. 6 were measured at the end of pyrolysis in nitrogen. Their overall loss amounts are 20%, 18% and 18%, respectively. Evidently, these weight changes are independent of the sample size and geometry. Data listed in Table 5 also demonstrate that the weight loss characteristics are not dependent on the sample geometry when heated in a nitrogen atmosphere.

Figure 18 shows the TG/DTG curve for a powder sample heated in a nitrogen atmosphere. The initial degradation temperature is 298°C, or about 50°c less than that of laminated fiber form. The powder sample removes gas diffusion processes. Sample size reduction promotes degradation. Hence, the thermal stability of the powder sample is lower than that of the laminated fiber sample.

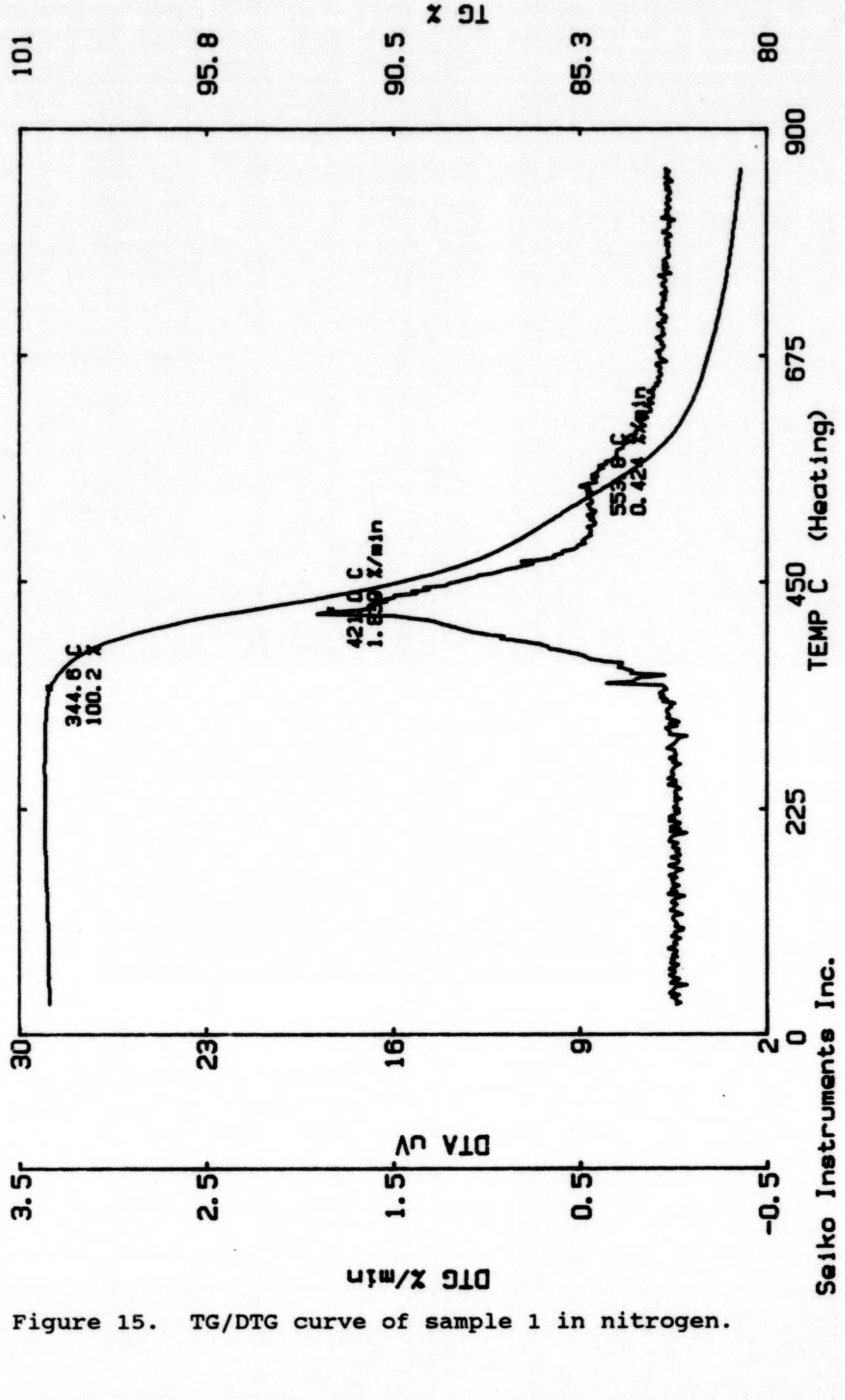

Figure 15. TG/DTG curve of sample 1 in nitrogen.

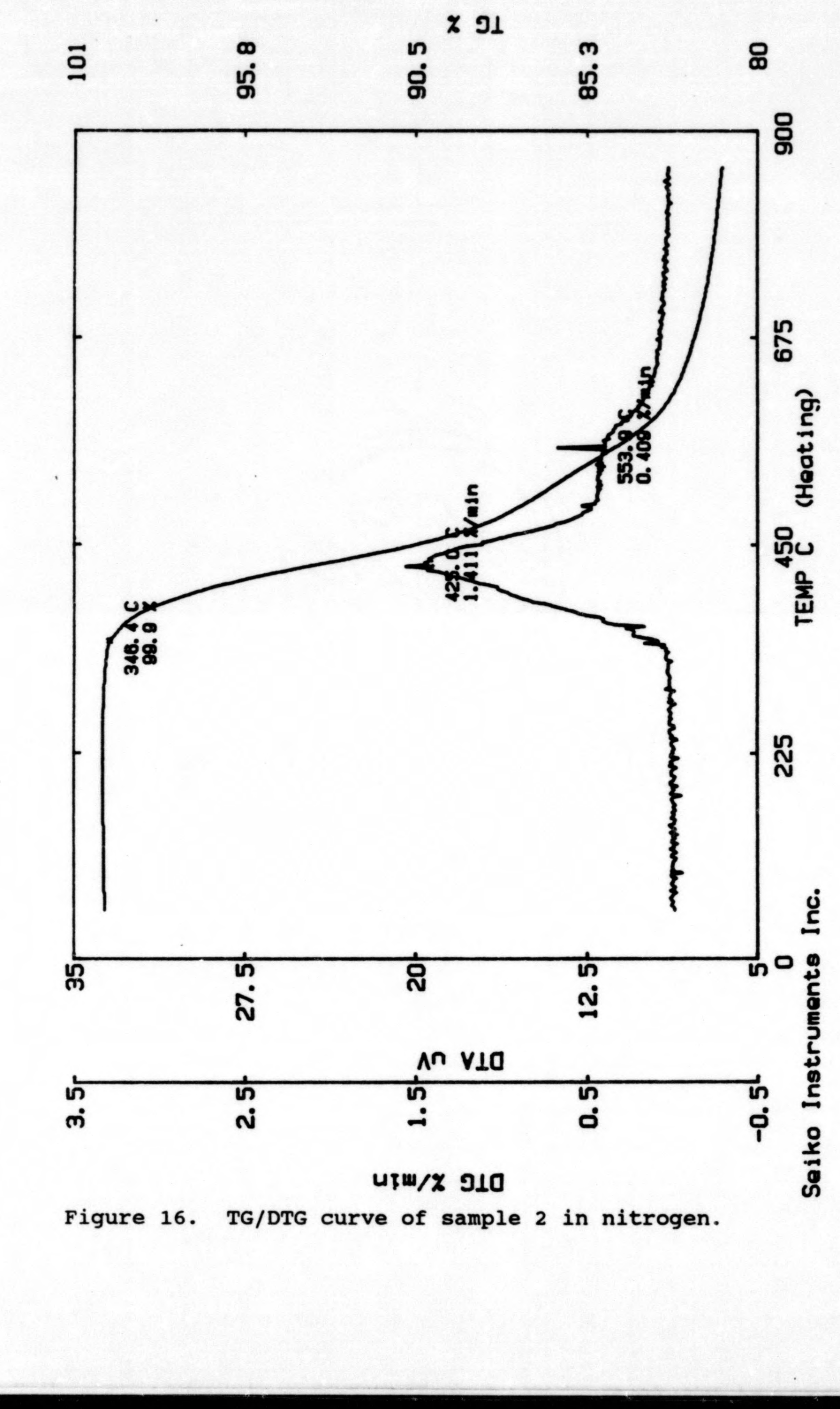

Figure 16. TG/DTG curve of sample 2 in nitrogen.

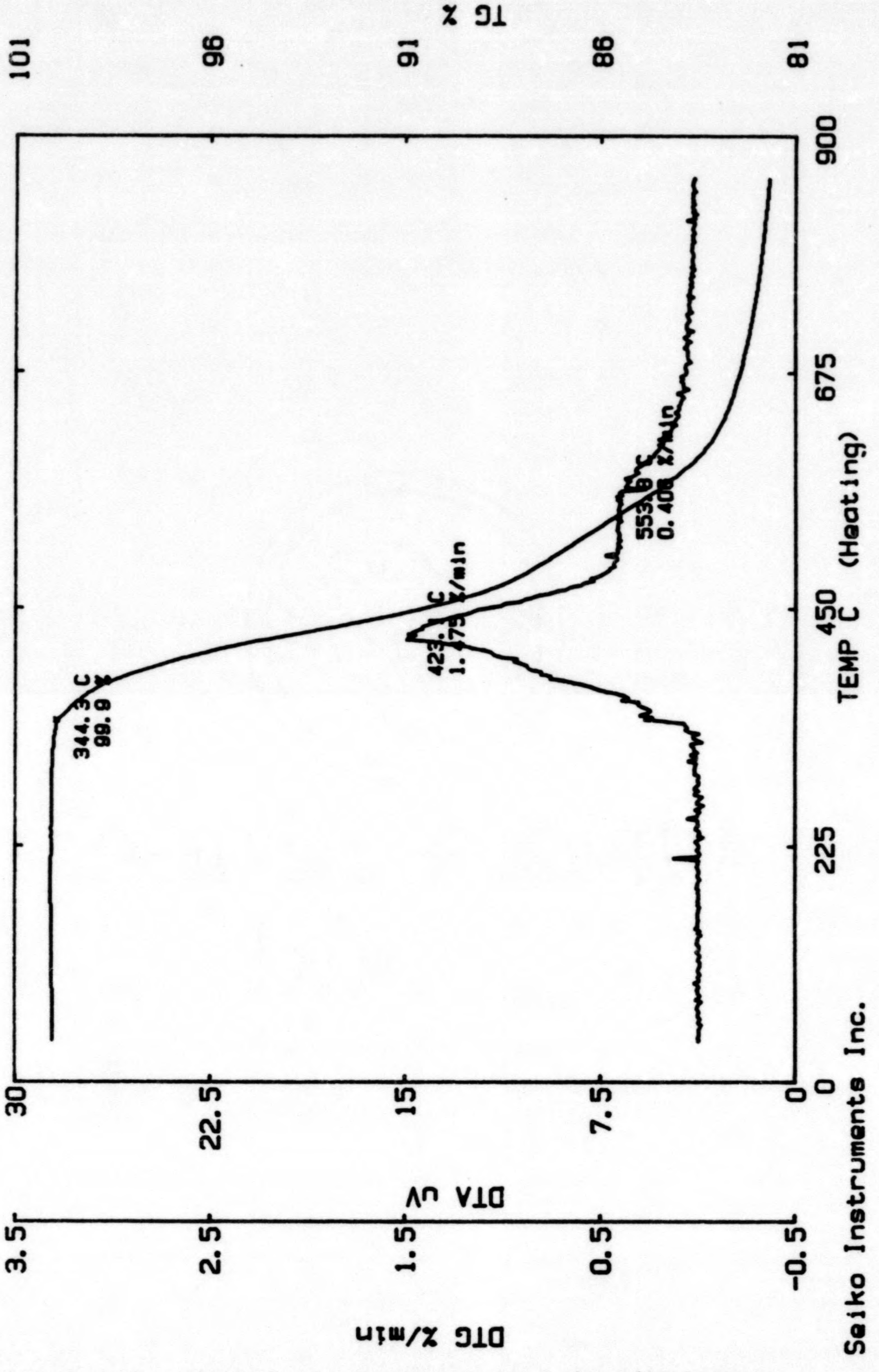

Figure 17. TG/DTG curve of sample 3 in nitrogen.

Table 5. TGA/DTG summary for samples 1-3 in nitrogen

Sample No.	T_i	T_{max1}	T_{max2}	W_1	W_2	W_3
1	347°C	419°C	554°C	5%	16%	19%
2	346°C	423°C	554°C	5%	16%	19%
3	346°C	421°C	554°C	5%	15%	18%

Parameters: T_i is the onset temperature of the initial degradation; T_{max1} is the temperature of the first maximum weight loss rate; T_{max2} is the temperature of the second maximum weight loss rate; W_1 is the percentage weight loss at T_{max1}; W_2 is the percentage weight loss at T_{max2}; W_3 is the percentage weight loss at 850°C.

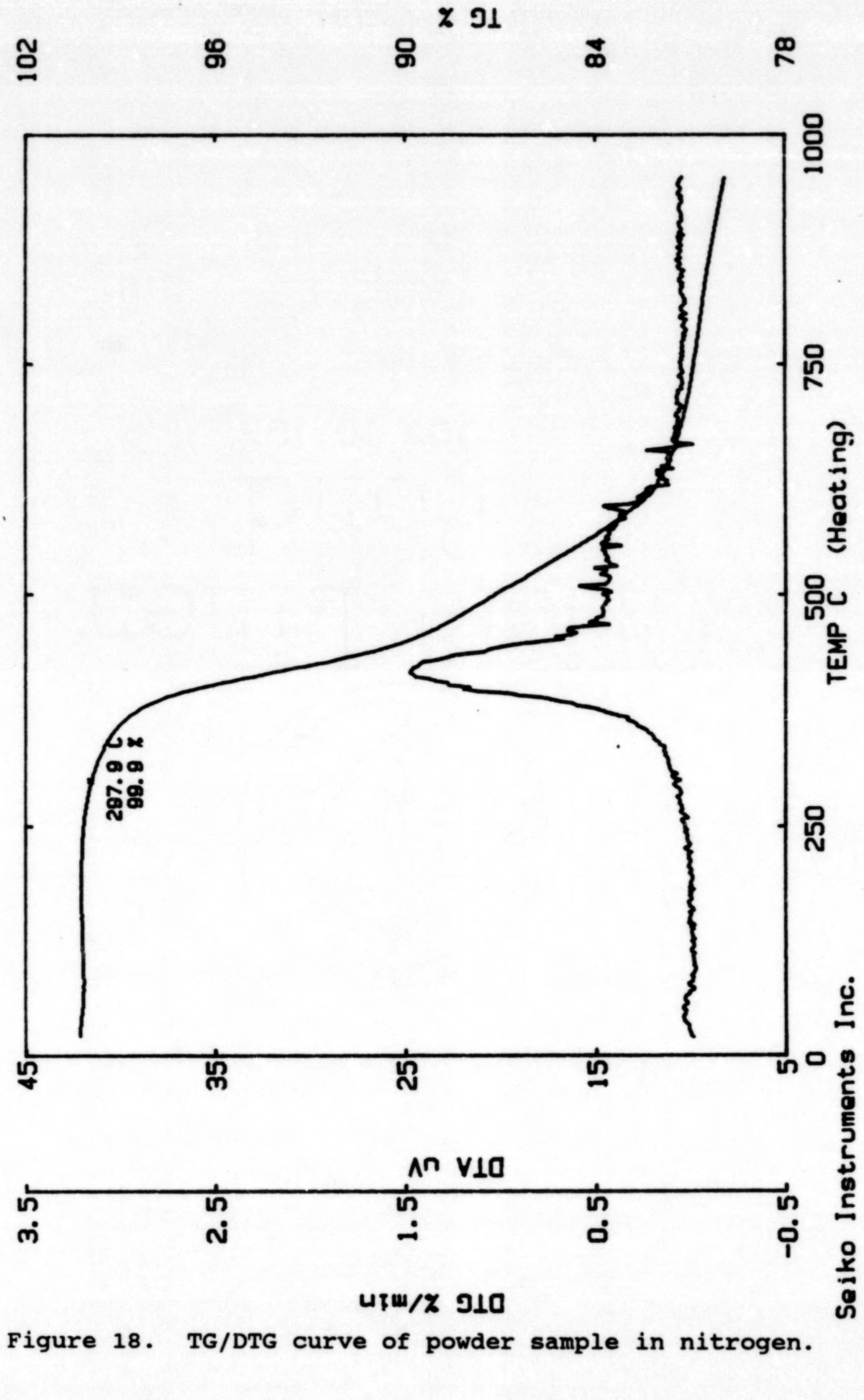

Figure 18. TG/DTG curve of powder sample in nitrogen.

II. TG/FTIR results

A typical FTIR spectrum, with IR band identification, of the evolved gases from the X5260/IM7 heated in air at 440°C is presented in Figure 19. The evolved gas products are composed of H_2O (1683 cm^{-1}), CO_2 (2359 cm^{-1}), CO (2110 cm^{-1}), NO (1920 cm^{-1}), CH_4 (3014 cm^{-1}), other aliphatic hydrocarbons (2969 cm^{-1}), and aromatic hydrocarbons (3086 cm^{-1}).[9,16,17] These evolved gas products also exhibited peak amounts around T_{max} (temperature of the maxima weight loss rate) as is illustrated in Figure 20. For instance, the amounts of H_2O, CO_2, aliphatic hydrocarbons, and aromatic hydrocarbons given off are relatively high at the first peak temperature of 440°C. Figure 21 shows the corresponding TG/DTG curve obtained from the DuPont 951 thermogravimetric analyzer in this TG/FTIR study.

A typical FTIR spectrum of the evolved gases during pyrolysis of X5260/IM7 at 430°C is shown in Figure 22. The evolved gas products are identified as CO_2, CO, CH_4, phenol, para-cresol, maleimide, succinimide,[11] other aliphatic hydrocarbons, and aromatic hydrocarbons. The pyrolyzates contain carbon monoxide, which suggests that two maleimide rings are most probably linked by an aromatic bridge in the X5260 resin system. The thermal degradation of polybismaleimide has been reported[11] to vary with the structure of the bridge between the succinimide rings. Aliphatic bridges are thermally less stable than the succinimide. Therefore the degradation proceeds preferably by

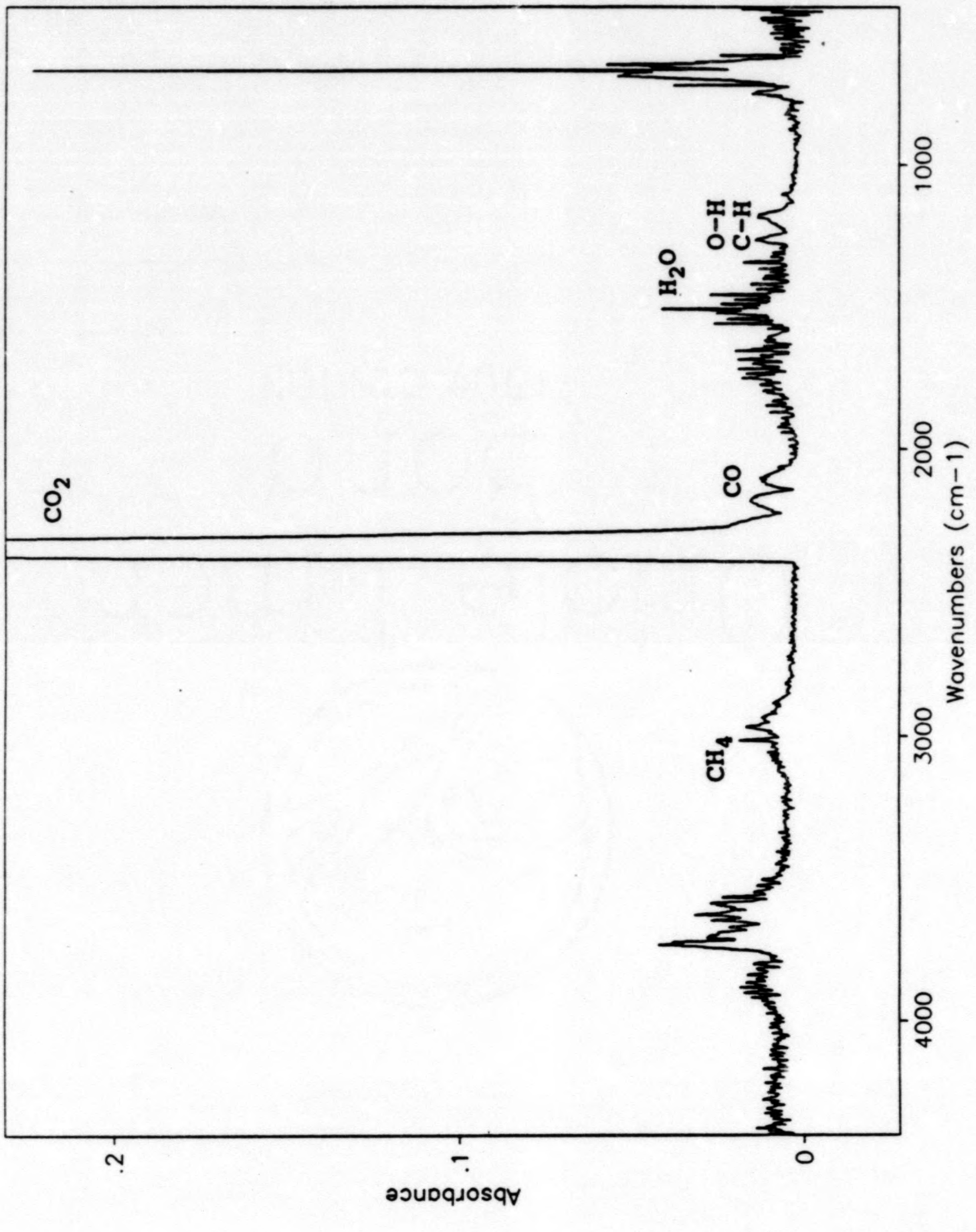

Figure 19. FTIR spectrum of evolved gases from X5260/IM7 heated in air.

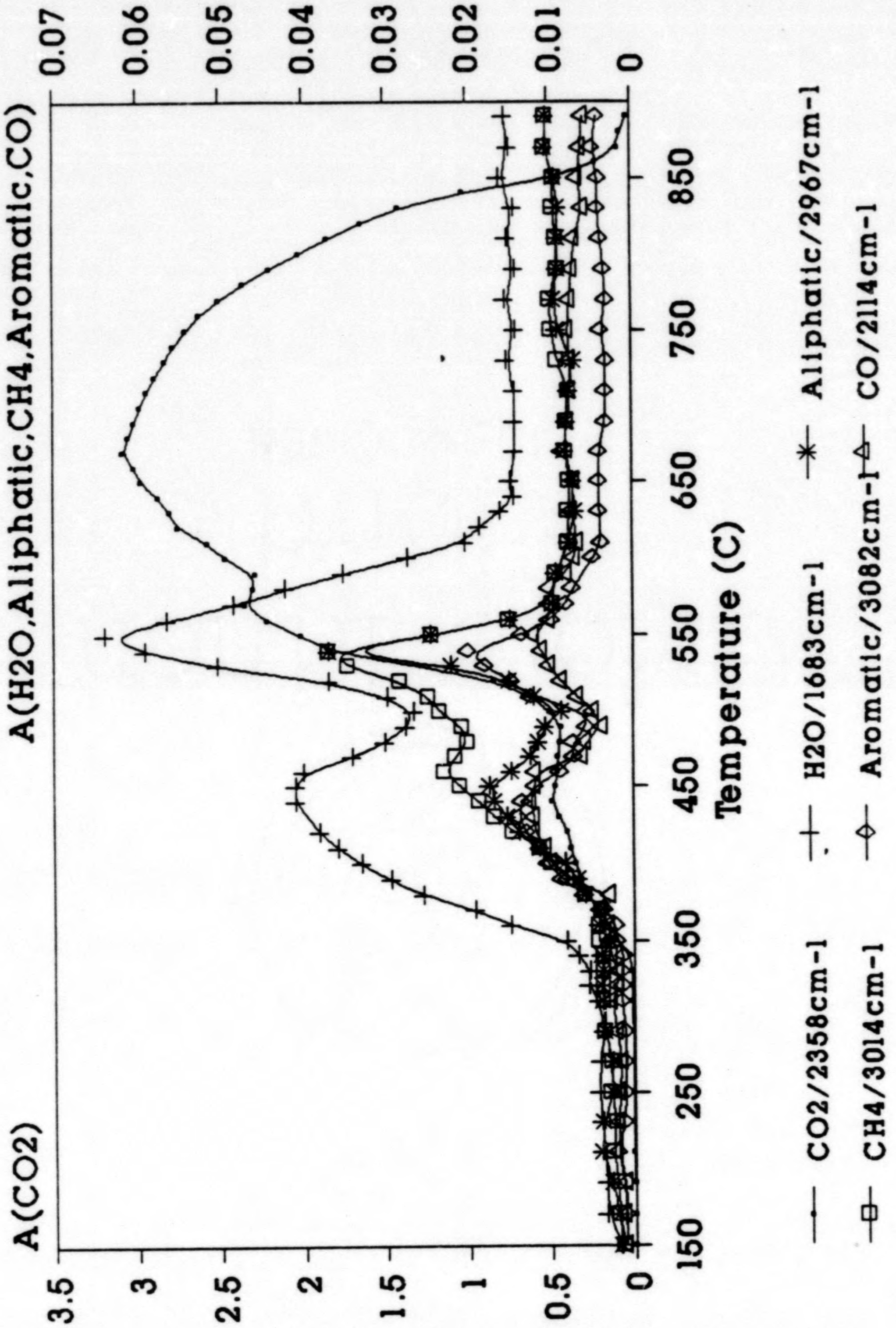

Figure 20. Gas release profiles during combustion of X5260/IM7.

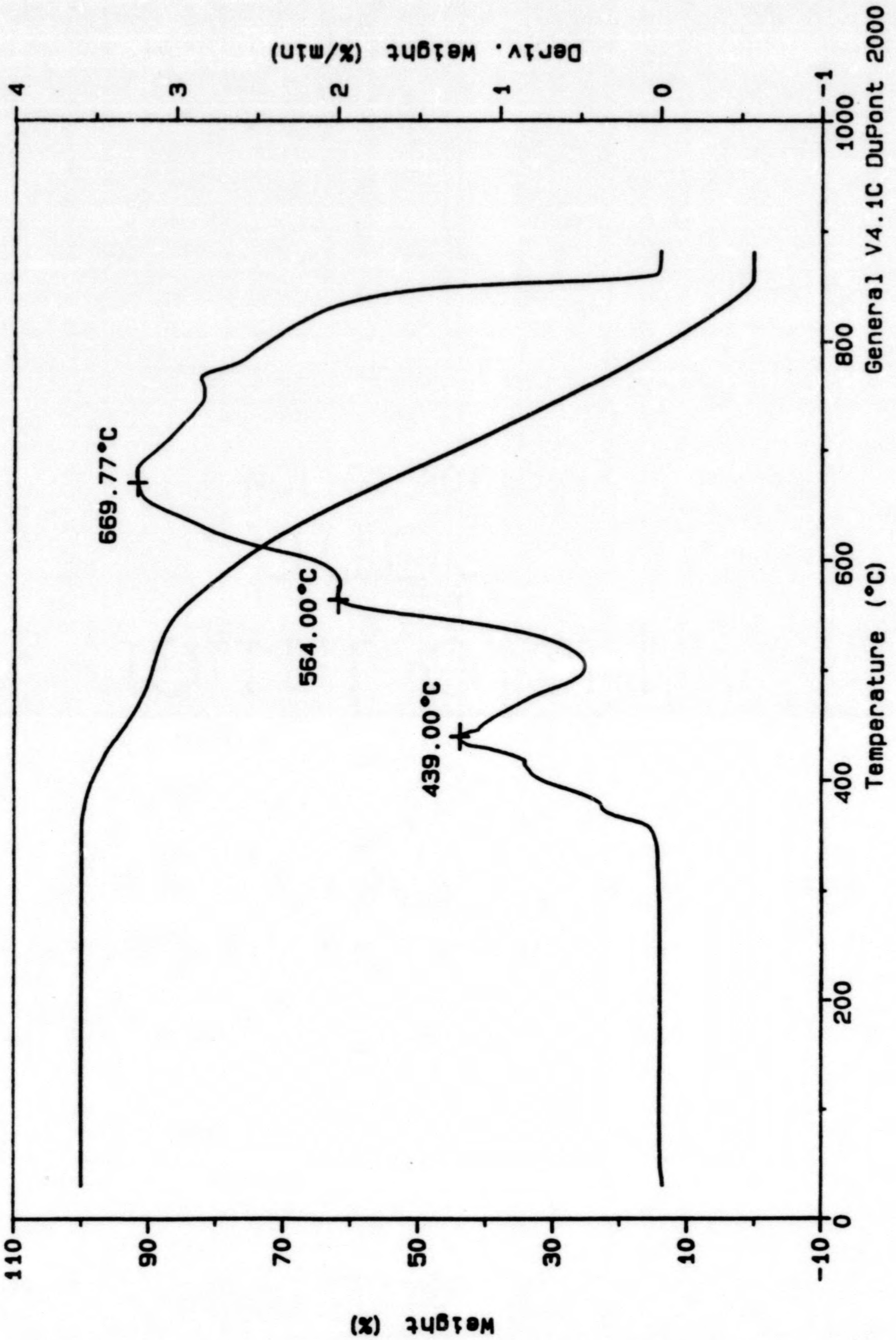

Figure 21. TG/DTG curve for heating X5260/IM7 in air for TG/FTIR study.

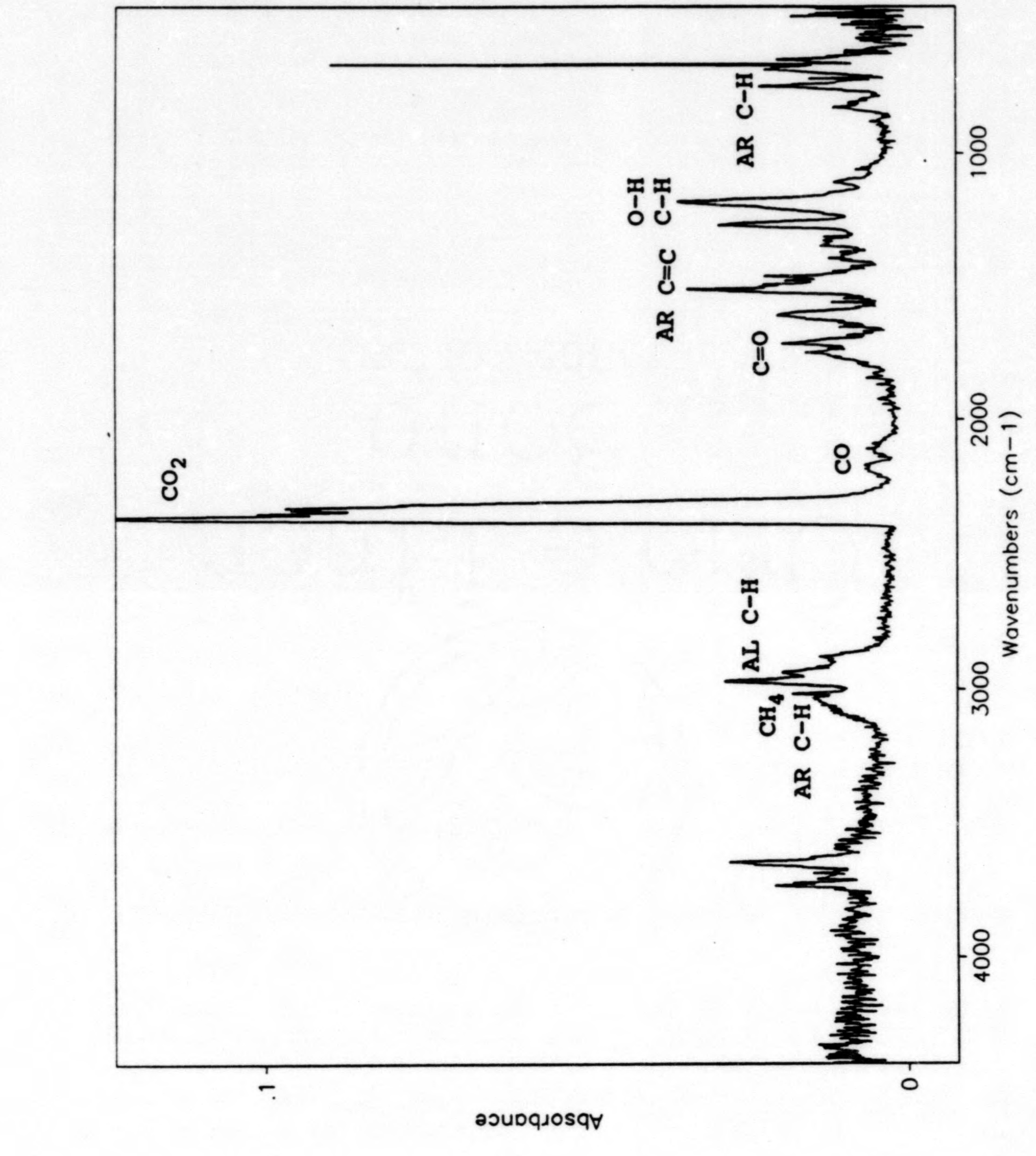

Figure 22. FTIR spectrum of evolved gases from heating X5260/IM7 in nitrogen.

cleavage of the bond between nitrogen and carbon, to be followed by succinimide abstraction. More thermally stable aromatic bridges cause succinimide ring breakdown, followed by carbon monoxide abstraction.

Table 6 provides a list of the wavenumbers of some functional groups occurring in organic compounds used to identify the species in the evolved gases during pyrolysis.[16] The TG/DTG curve for the TG/FTIR study of heating X5260/IM7 in a nitrogen atmosphere is shown in Figure 23. The corresponding gas release profiles (Figure 24) also show two major peaks around the maximum weight loss rate peak temperatures. At 430°C, relatively high amounts of evolved CO_2, aliphatic hydrocarbons, and aromatic hydrocarbons are evident. The amount of evolved CH_4 is relatively high at 560°C.

Table 6. Wavenumbers of some functional groups in organic compounds selected to identify the species in the evolved gases during pyrolysis

Functional Group	Wavenumber
O-H (stretching)	3647 cm^{-1}
aromatic C-H, aliphatic C-H (stretching)	2880-3060 cm^{-1}
C=O (stretching)	1715, 1748 cm^{-1}
aromatic ring (C=C vibrations)	1507, 1607 cm^{-1}
alkane CH_2 (scissoring) and CH_3 (asymmetric bending)	1460 cm^{-1}
CH_3 (symmetric bending)	1373 cm^{-1}
C-H (bending), O-H (bending)	1340, 1176 cm^{-1}
C-N, C-O, C-OH (stretching)	1100-1300 cm^{-1}
aromatic C-H (out-of-plane bending)	747, 828 cm^{-1}

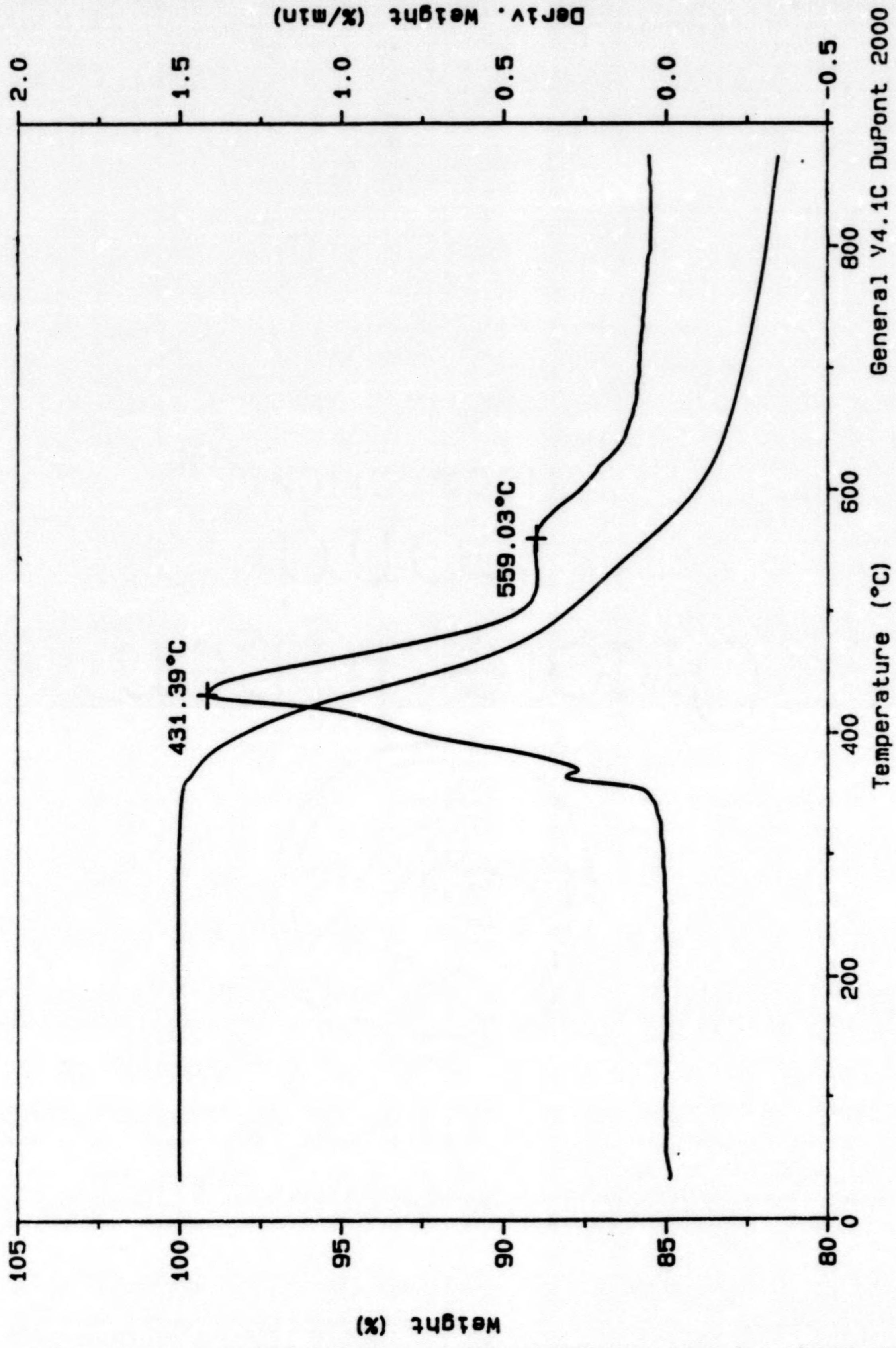

Figure 23. TG/DTG curve of X5260/IM7 heated in nitrogen for TG/FTIR study.

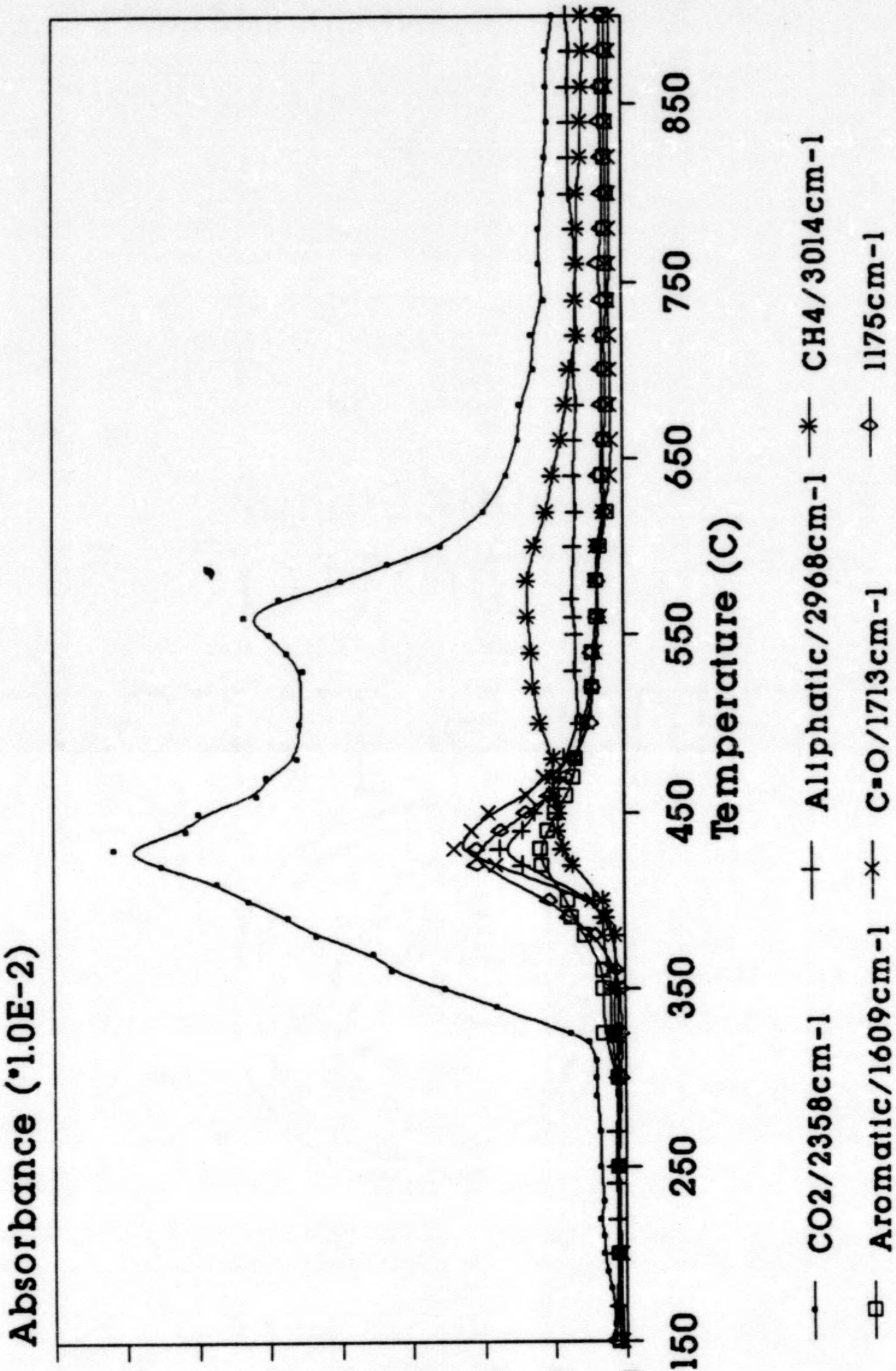

Figure 24. Gas release profiles during pyrolysis of X5260/IM7.

CONCLUSIONS

Dynamic TG studies of the composite X5260/IM7 indicated that the composite began to lose weight at approximately 352°C in air atmosphere. Sample one lost 5% and 10% of its weight at 421°C and 457°C, respectively. Its weight loss rate typically exhibited three maxima at 421°C, 582°C and 657°C, and the corresponding weight losses are approximately 5%, 24% and 54%. These weight loss characteristics were found to depend upon test specimen thickness and surface area-to-volume ratio due to gas diffusion. At the end of the 10°C/min TGA run, very little of the composite remained except for the large and thick sample tested.

In nitrogen, the composite began to loss weight at approximately 345°C. Its weight loss rate showed two maxima. These weight loss characteristics were observed to be independent of sample thickness and surface area-to-volume ratio.

The TOS of composite X5260/IM7 in the 3% oxygen atmosphere was examined to simulate aircraft flight environment, a thermal oxidative stability higher than that in air was observed, as expected.

The laminated fiber form of the composite is thermally more stable than the powder form.

Gas cell FTIR showed gas products given off to be CO_2, CO, NO, H_2O, CH_4, phenol, and other aliphatic and aromatic hydrocarbons. It was interesting to note that these gas products also exhibited peak amounts of evolution at weight loss rate peak temperatures.

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