

APPLICATION NOTE

Polymers – Differential Scanning Calorimetry

Failure Analysis by Means of DSC – Case Study: Breakage of Nylon Ties

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Introduction

Nylon ties are produced by injection molding using UL-approved PA66 (Nylon 66). Due to its good acid resistance, corrosion resistance, isolation and mechanical properties, and aging stability, nylon ties are widely used in electronic and electrical appliances, wires and cables, lamps and lanterns, machinery and equipment, ship-building, construction, and other industries.

Breaking strength is one of the most important performance indicators of nylon ties, whether the fracture occurs in the end part, center, or reverse teeth, the fracture strength should be higher than the nominal value of the tension strength. When the fracture strength is less than the nominal value, one of the solutions is to replace the current tie with another product with higher tension strength. However, the possible reasons for failure of

the nylon tie should be considered, e.g., high temperature and long storage time results in aging and embrittlement. But also, the loss of moisture will significantly influence the mechanical properties. A fundamental cause can always be replacement of the original material with low-cost raw materials or the usage of recycled materials. Last but not least, the processing conditions have important influence on the final quality of the ties.

Sample Preparation and Measurement Conditions

In this application note, the causes of breakage were analyzed by the DSC method; the details are as follows: There are 3 nylon tie samples, which are sample #1 OK, sample #2 stock, and sample #3 NOK (broken in the middle), as shown in figure 1. The measurement conditions are detailed in table 1.



1 Photo of the samples

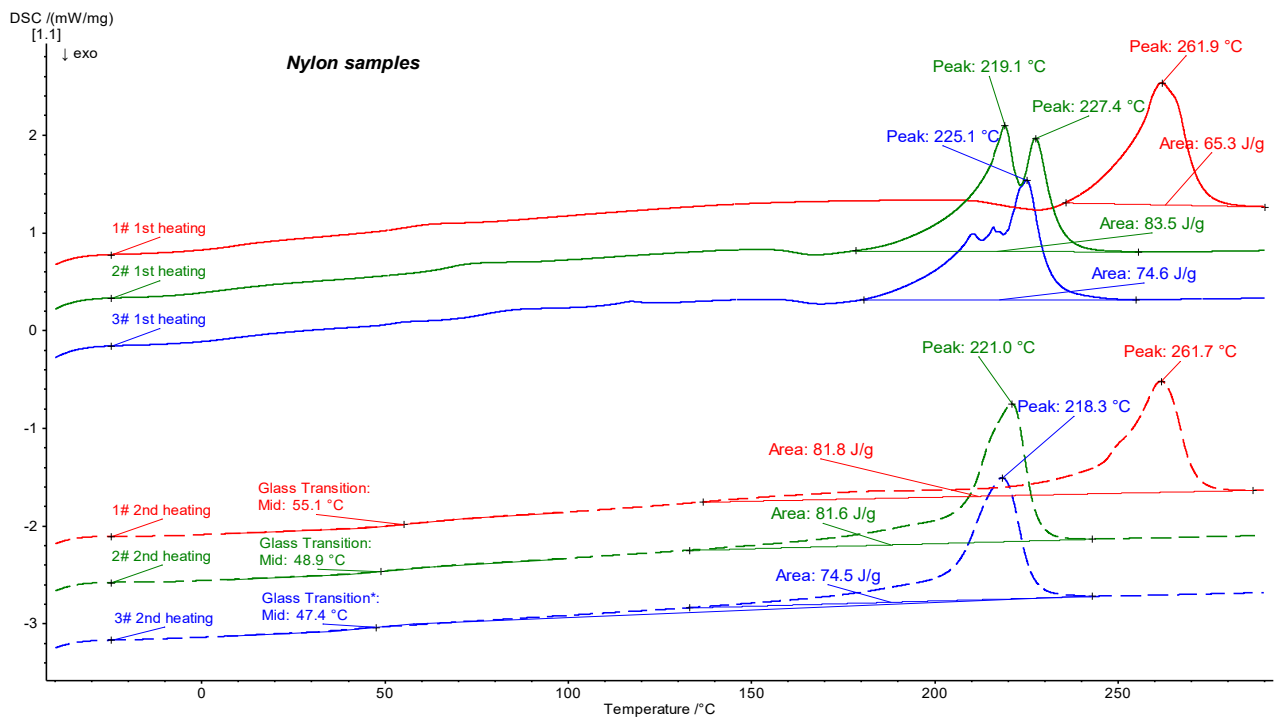
Table 1 Parameters for DSC measurements

Sample	Sample #1 OK	Sample #2 Stock	Sample #3 NOK
Sample mass [mg]	9.48	9.03	9.04
Crucible	Concavus® Al crucibles, pierced lid		
Temperature program	-50°C ... 300°C		
Heating/cooling rate	20 K/min		
Atmosphere	Nitrogen (20 ml/min)		

Measurement Results

For comparison of the differences of the three samples, the heating curves and cooling curves are shown in figures 2 and 3, respectively. Sample #1, sample #2 and sample #3 are presented in red, green and blue.

According to the results of the 1st heating, the melting temperature (peak 261.9°C) of sample #1 is in the range of the theoretical melting temperature of PA66 (225°C to 265°C). The melting temperature ranges of samples #2 and #3 are quite similar, although the shapes of the peaks are different. The shape of the first heating curve is influenced by the thermomechanical history (such as processing conditions but also sample preparation), and therefore, this will not be discussed further.



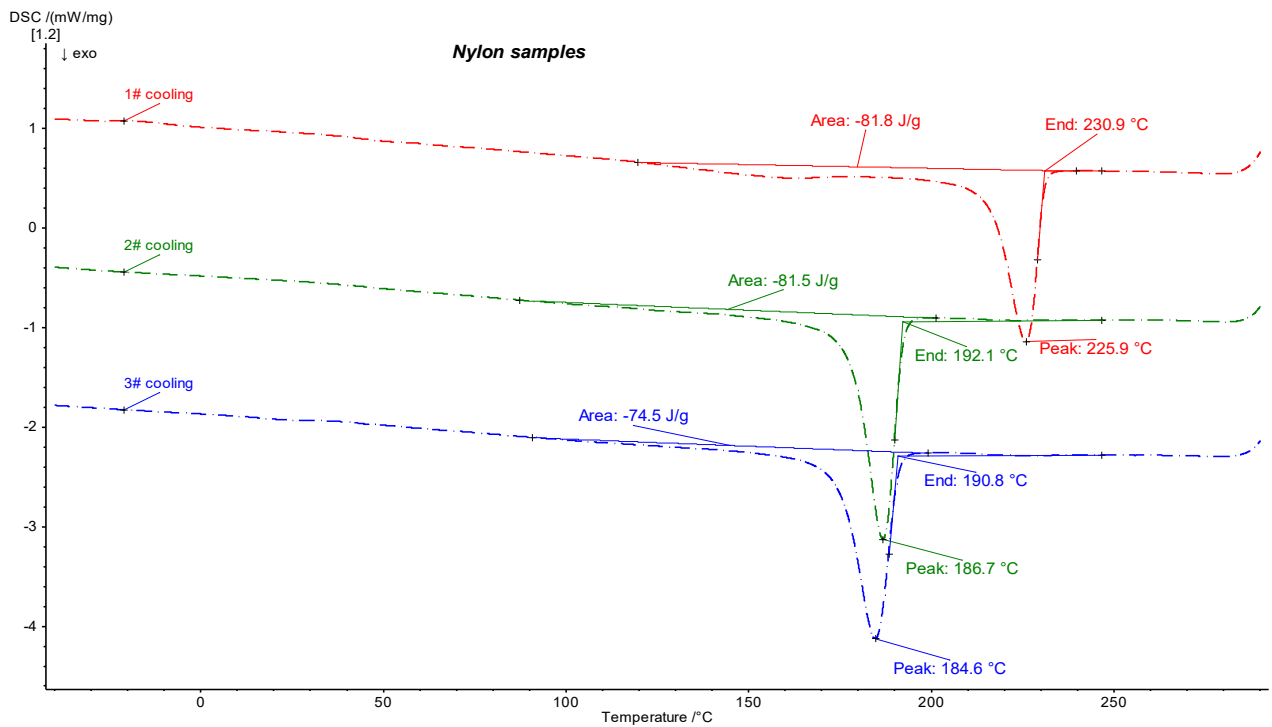
2 DSC results for the 1st heating curves (upper curves) and for the 2nd heating curves (lower curves)

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For direct comparison of the material, it is better to compare the 2nd heating curves, as the thermal history of all samples is now exactly the same. The melting peaks of samples #2 and #3 are quite similar, which means the compositions of sample #2 and sample #3 are most likely the same. Sample #1 shows completely different melting behavior. It's therefore supposed that sample #1 must be made from a different material.

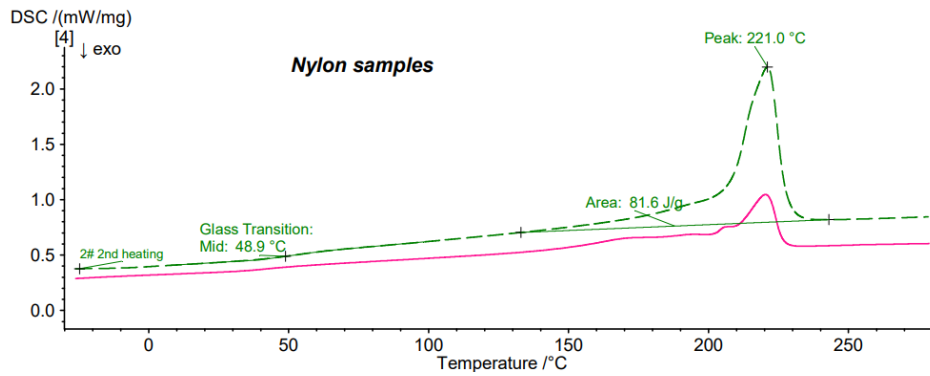
Combining the results of the 2nd heating and cooling, it can be presumed that sample #1 is likely PA66. The material of samples #2 and #3 is quite similar and it may be another type of polyamide.

The cooling curves show that the crystallization temperature of sample #1 (peak 225.9°C) is higher than that of the other 2 samples, and the crystallization temperatures of samples #2 and #3 are similar.



3 DSC results for the cooling curves

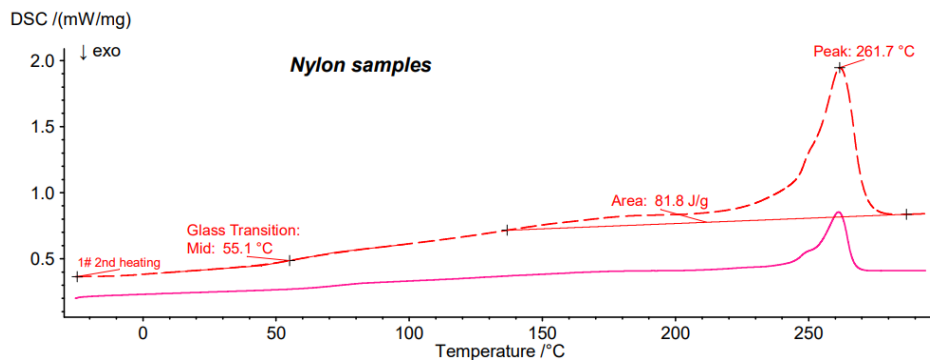
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Results:

Database Entry	Similarity
PA610_DSC	98.92 %
PA612_DSC	96.13 %
PA6-GF30_DSC	93.96 %
PBT_DSC	86.18 %
PVA_DSC	78.85 %
PVF_DSC	67.69 %

4 Identify results of sample #2



Results:

Database Entry	Similarity
PA66-GF30_DSC	86.48 %
ETFE_DSC	83.87 %
PET_DSC	69.43 %
PA46_DSC	55.65 %
PPS_DSC	54.07 %
PU_crosslinked_DSC	53.55 %

5 Identify results of sample #1

To identify the material of samples #2 and #3, the *Identify* function of the *Proteus*® software was used. The similarity between the 2nd heating curve of the #2 sample and PA610 reaches 98.92% (the green curve in figure 4 is the 2nd heating curve of the #2 sample, and the red curve is the standard curve of PA610 in the database), which indicates that the material of the #2 and #3 samples is likely PA610.

Figure 5 shows the *Identify* result of the 2nd heating curve of sample #1; the similarity between sample #1 and PA66 in the KIMW database* is almost 90%, which confirms the previous assumption.

Summary

According to the results of *Identify*, sample #1 OK is made of PA66, but sample #2 stock and sample #3 NOK are made of PA610. The mechanical properties of PA66 and PA610 are different, and the melting and crystallization behavior is not the same, even if they were processed with the same method; one of them might be overheated or degraded, which may influence the performance of the products – for example, it may easily fracture.

*KIMW database for DSC measurements on polymers, a collaboration with Kunststoffinstitut Lüdenscheid, Germany.