

# Fusing Characteristics of Topas<sup>®</sup> COC Based Toner

*Klaus Berger\*, Doug Hammond<sup>#</sup> and Toru Nakamura<sup>+</sup>*

*\*Research Chemist, Ticona GmbH, Building G, Frankfurt, Germany*

*<sup>#</sup>Development Engineer, Ticona Polymers Inc., Burbank, California, USA*

*<sup>+</sup>Director / Marketing Manager, Ticona Japan Ltd., Bunkyo-ku, Tokyo, Japan*

## Abstract

In this study the impact of toner binder resin design, especially molecular weight and molecular weight distribution, on the fusing performance in oil-free hot roll fusing has been studied for the new class of environmentally friendly cyclic olefin copolymers (COC). The first results presented in this report indicate that the fusing latitude, or so-called anti-offset-window (AOW), in hot roll fusing is strongly influenced by the amount of longer chains in the underlying binder resins. Surprisingly, only the hot offset temperature increases with the amount of longer chains in the binder resin, whereas the cold offset temperature is not affected at all. In parallel with a larger AOW, the minimum temperature for acceptable fixing is increased slightly, and lower image gloss is obtained.

More detailed studies on a variety of exemplary toner formulations are necessary to better understand the impact of binder resin design; T<sub>g</sub>, M<sub>n</sub>, M<sub>w</sub>, molecular weight distribution, and elasticity effects on fusing performance need to be explored.

## Introduction

Today electrophotographic toner materials offering low minimum fusing temperature, wide fusing latitudes, improved charging properties, excellent pigment and wax dispersion, high jettability, and low price are required in the market.<sup>1</sup> Dry xerographic toner essentially consists of a binder, a colorant and various other ingredients like magnetite, charge control agents (CCA), waxes, surface additives and other components.<sup>2</sup> The role of the polymer is to bind the pigment to the paper and form a permanent image. Toner fusing properties like minimum fusing temperature and fusing latitudes, are known to be dominated by the physical and chemical properties of the binder resin.<sup>3</sup> One approach to achieve the demanding goals set by printer manufactures is by optimizing the performance of the currently employed polyester or styrene-acrylic resins, e.g., by introducing crystalline polymers or waxes into the principal polyester binder, or by introducing new functionalized monomers into styrene acrylics, as described

in recent publications.<sup>4,5</sup> Another promising approach is offered by the use of cyclic olefin copolymers (COC), a new class of polyolefin resins developed and introduced under the Ticona trade name Topas<sup>®</sup> COC. According to recent publications, these materials offer benefits over traditional binder resins, especially when high printing speed, stable tribocharging and environmentally friendly materials are required.<sup>6-8</sup> Today two COC grades for use as toner binders are commercially available, a low molecular weight polymer with narrow molecular weight distribution for non-contact fusing – Topas<sup>®</sup> COC TM, and a bimodal material for hot roll fusing called Topas<sup>®</sup> COC TB.<sup>9</sup>

These two materials form completely homogenous mixtures by ordinary melt processing, thus providing a way to tailor and optimize the fusing properties of the toner to the requirements of the printer and fuser unit. In this study, the effect of toner binder resin design, especially molecular weight and molecular weight distribution, on the fusing properties like minimum fusing temperature, hot offset temperature and anti-offset-window was measured for model toners. Furthermore, basic image properties like fixing strength and gloss levels as a function of fusing temperature are reported and compared to other commercial toners.

The goal of this study is to describe the impact of binder composition on physical properties and fusing performance, thus supporting printer and toner manufacturers in their development work and providing resin manufacturers direction for future improvements. It is not the goal of this study to supply a toner showing optimum performance in a particular printer and copier. That is usually best achieved in close collaboration between toner manufacturer and raw materials supplier.

## Experimental

Figure 1 describes the applied toner preparation and testing procedure.

A standard toner formulation comprising 7% pigment, 2% CCA and 7% wax for 100% of binder resin was used within the present study to ensure that the observed effects are only caused by changes of the binder resin and not by changes of the other toner ingredients.

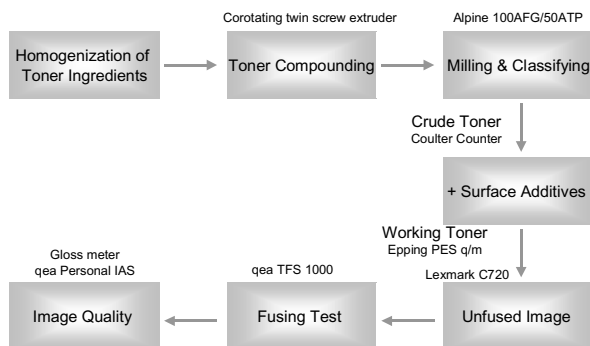


Figure 1. Applied toner preparation and testing procedure.

At the beginning all ingredients were crushed and premixed to provide good homogenization. Binder mixtures were prepared in a pre-compounded form.

The toners were prepared on a co-rotating twin-screw extruder under appropriate processing temperatures in the range of 120 – 200°C. The quality of the pigment dispersion was observed with an optical microscope. As necessary, quick adjustments of the processing conditions were made to optimize dispersion. The compounded mixture was then milled to a volume-averaged diameter of about 8.5  $\mu\text{m}$ . Finally, the toner was classified to get rid of excess fines by using a standard Hosokawa Alpine 100AFG/50 ATP. The physical properties of these samples were then characterized.

Afterwards the toner was transformed into a working toner by surface addition with about 0.6 wt% of a standard fumed silica. This permitted adjustment of the tribocharge properties and improved the powder flow. To prepare unfused images, a Lexmark C720 non-magnetic monocomponent color laser printer with a disabled fusing unit was used. The toner was put into carefully cleaned toner cartridges, and the transferred toner mass/area of monochrome test targets was measured in comparison to the OEM toner.

The fusing performance was studied with a test target composed of three monochrome squares in a row on a TFS 1000 fusing rig available from Quality Engineering Associates, Inc. (qea). A roller speed of 125 mm/s, a paper feed rate of 120 mm/s, and a nip pressure of 15 psi were employed. The fusing tests were done on 80 g/m<sup>2</sup> paper, without added fuser oil. In a standard test procedure, the fusing performance was measured for each temperature with three printed pages separated by two blank pages. During the experiments the real temperature of the fusing roll was measured, as it normally drops during fusing experiments. Fusing tests were performed in a set temperature range from 130 to 240°C at increments of 5°C.

Finally, the fixing ratio was measured by comparing the ratio of optical image density (OD) before and after folding with the Personal IAS of qea. Average values of at least 5 measurements on every square are reported. In addition, gloss measurements at 85° observer angle are presented.

For better clarity the following discussion is limited to only two exemplary Topas® COC-based toner formulations in comparison to the performance of Lexmark OEM toner.

## Results and Discussions

### Basic Physical Toner Properties

The physical properties of the studied toner samples are shown in table 1.

Table 1. Basic Physical Properties of Studied Toner Samples.

Sample	Basis	T <sub>g</sub> / °C	T <sub>1/2</sub> / °C	T <sub>1/2</sub> – T <sub>fb</sub> / °C
1	OEM	64	124	22
2	COC	58	127	20
3	COC	59	154	28

The glass transition temperature T<sub>g</sub> was measured by DSC on a TA 2920 at 20 K/min in the second heat as the temperature where half of the step-change in heat flow occurred. The flow test was measured by using a Shimadzu CFT-100D flow tester at 8 kg load, a 1 x 10 mm die and 5 K/min heating rate.

According to Table 1, the OEM toner 1 shows high melt flowability and a standard T<sub>g</sub> value for toner in the range of 60°C. Additional IR and GPC (gel permeation chromatography) measurements reveal that a low molecular polyester binder resin is used.

The other two toners are based on COC. Toner 3 is totally based on Topas® COC TB available from Ticona, while a 50/50 mixture of Topas® COC TB and TM was used for Toner 2. The T<sub>g</sub> values of both toners are comparable, but the half-flow temperature T<sub>1/2</sub> is significantly lower for Toner 2. By incorporating 50% of the high flow, low molecular weight Topas® COC TM material, melt flow is greatly enhanced. In parallel, the temperature difference between T<sub>1/2</sub> and beginning-flow temperature T<sub>fb</sub> is significantly smaller for Toner 2. This demonstrates a more pronounced temperature dependence of the melt viscosity, thus indicating a smaller fusing latitude.

The difference in composition of Toners 2 and 3 also are demonstrated in their rheological behaviors. This was studied with so-called “frequencies sweeps” at different temperatures on a Physica MCR 300, plate-plate geometry rheometer, from low to high temperatures to better simulate the fusing condition in a printer. For a better comparison, universal master curves at an intermediate temperature of 150°C were constructed by applying the time-temperature superposition principle. The master curves for the storage modulus G' and the loss modulus G'' as a function of reduced frequency are shown in Figure 2.

According to Figure 2, the terminal flow region characterized by a slope of –1 for G'' and –2 for G' in this double logarithmic plot occurs at significantly higher reduced frequencies in the case of Toner 2. Simultaneously, the so-called rubbery plateau region with an almost constant value of G' extends over a larger frequency range for Toner 3, demonstrating a higher number of entangled chains, and consequently an elevated melt strength.

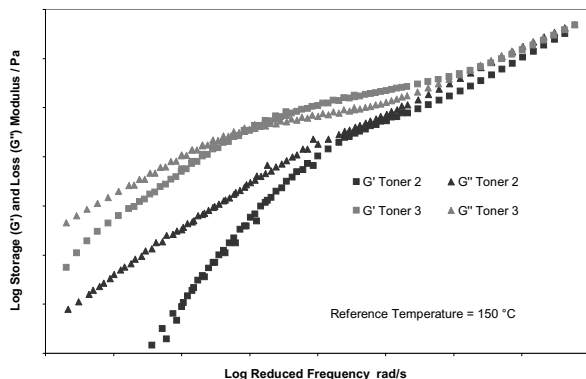


Figure 2. Viscoelastic properties of toner 2 and 3.

### Fusing Properties

The transferred toner mass / area for the test pattern was  $3.9 \mu\text{g}/\text{mm}^2$  for the OEM toner and  $3.4 \mu\text{g}/\text{mm}^2$  in the case of the COC toners. Taking into account the different mass densities of  $1.25 \text{ g}/\text{cm}^3$  for polyester and  $1.00$  for COC, this translates into a somewhat higher pile height of  $4 \mu\text{m}$  for COC compared with  $3 \mu\text{m}$  for the OEM toner.

The temperature range where no visible offset under the employed test conditions occurred, the so-called anti-offset-window (AOW), is given in Table 2.

Table 2. Fusing Properties of Studied Toner Samples.

Sample	Basis	No cold / °C	No hot / °C	AOW / °C
1	OEM	no	no	0
2	COC	145	170	25
3	COC	145	195	50

As seen in Table 2, the Lexmark OEM toner shows offset whether cold or hot offset over the whole temperature range under the employed fusing conditions. Consequently the AOW is  $0^\circ\text{C}$ . In order to counterbalance the insufficient fusing properties of the pure toner, a high amount of fuser oil is used in the actual printer to achieve an acceptable fusing latitude.

In the case of the COC-based toner, acceptable anti-offset properties are observed starting at about  $145^\circ\text{C}$ . As estimated from the viscoelastic properties, a wider fusing latitude of  $50^\circ\text{C}$  in comparison to  $25^\circ\text{C}$  is observed for Toner 3. Table 2 further demonstrates that the gain in AOW is entirely caused by changes of the hot offset temperature, reflecting the elevated melt strength of Toner 3. Surprisingly the cold offset temperature is not affected at all by the resin composition. This observation should be validated by additional experiments.

### Basic Image Quality

With the present test target, an initial image density of  $1.05$  was reached for the Lexmark OEM toner, while the COC toner showed a value of  $0.8$ . This lower initial image

density of the COC toner, in contrast to the comparable transferred mass, may be explained by higher pigment content and/or better pigment dispersion in the OEM toner.

Figure 3 depicts how the fusing strength, determined as the optical density OD in the crease, compared to the initial OD before folding changes as a function of fusing temperature. The temperature where no cold-offset is observed is marked by a large symbol.

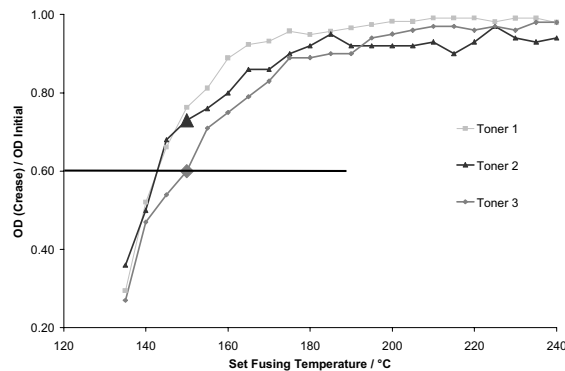


Figure 3. Fusing strength as a function of  $T$  for studied toner.

As shown in Figure 3, all the toner samples exhibited comparable fusing strength as a function of temperature:

- At low fusing temperature loose fusing is observed, while the fixing strength increases dramatically with increasing temperature.
- At higher fusing temperature the fixing strength slowly reaches its limiting value of  $1$ .

As further indicated in Figure 3, fusing at low temperature is best for the OEM toner, and decreases from Toner 2 to 3. Yet the shift of the curves is less than  $10^\circ\text{C}$  in fusing temperature for these two extreme samples.

If a fusing strength of  $0.6$  is set as the lower limit for acceptable fusing, such fusing is easily achieved if cold offset is avoided.

The change of image gloss measured at  $85^\circ$  with real fusing temperature is presented in Figure 4.

It is obvious from Figure 4 that image gloss is highest for the OEM toner and decreases from sample 2 to 3. Furthermore, the gloss for a given toner sample increases more or less linearly with respect to fusing temperature.

High gloss levels are caused by a smooth toner surface after fusing. Consequently, high image gloss is expected for toner samples showing high melt flow or low melt viscosity. The observed gloss levels are thus easily explained from the melt flow data shown in Table 1 and the viscoelastic properties contained in Figure 2. As melt viscosity decreases strongly with increasing temperature, gloss increases as well. Still, it is quite surprising that the increase of gloss with temperature is less significant than with the decrease in melt viscosity.

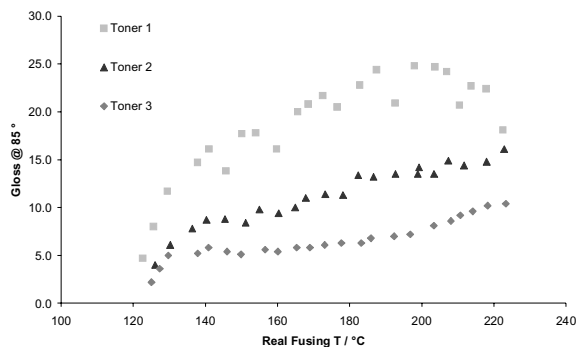


Figure 4. Image gloss as a function of  $T$  for studied toner.

## Conclusion

In this report, the impact of molecular weight design on fusing properties was studied for exemplary COC toner formulations. Compared with OEM toner based on low molecular weight polyester, the COC materials show a descending fusing latitude in oil-free hot roll fusing. The observed differences in fusing latitude can be explained by the molecular weight design of the binder resin system and their rheological behavior. Thus the test protocol used seems to deliver reliable results. Surprisingly, only the hot offset resistance, and not the cold offset temperature, is influenced by a higher amount of longer chains in the binder resin.

More detailed studies on a variety of exemplary toner formulation are necessary to better understand the impact of binder resin design.  $T_g$ ,  $M_n$ ,  $M_w$ , molecular weight distribution and elasticity effects on fusing performance are all expected to play a role in binder performance, and will be explored. Such a study will then allow further optimization of the toner systems using COC resins.

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## Biography

**Klaus Berger** received his Ph.D. in Physical Chemistry from the university of Paderborn, Germany, in 1995. He then worked on biodegradable polymers at the Federal Institute for Cereal, Potato and Starch Research in Detmold, Germany and on the rheological properties of associating polymer solutions at the Laboratory for Ultrasounds and the Dynamics of Complex Fluids in Strasbourg, France. In 1997 he joined the research and development group of Topas® COC within Hoechst and later Celanese/Ticona. He is currently working on Topas® COC toner binder resins for high quality printing in a fully equipped state-of-the-art polymer and toner lab. He is a member of IS&T.