

## A SYNCHROTRON STUDY OF POLYMORPHIC PHASE TRANSITION IN NYLON 6

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Aliphatic polyamides, such as nylon 6 (polyamide 6) are well known for their strong hydrogen bonding ability. The hydrogen bonds are the driving force that locks the crystallizing lamella into one or another crystalline form, satisfying a requirement of complete hydrogen bonding. Hydrogen bonds are also present in the amorphous regions and even in the molten state.

There exist two well resolved and documented crystal structures of PA6, *i.e.* the  $\alpha$  and  $\gamma$  forms. It is generally accepted that the monoclinic  $\alpha$  form is the thermodynamically most stable and that it is present after cooling slowly from the melt or annealing at high temperatures [1]. The monoclinic  $\gamma$  form, obtained by a treatment with an iodine-potassium iodide solution is also very stable and does not easily convert to the  $\alpha$  form [2]. Fast cooling or cold crystallization promotes development of a (pseudo)hexagonal  $\beta$  structure which, depending on cooling rate, can coexist with the  $\alpha$  form [1,3]. Most polyamides undergo a reversible transition between the pseudo-hexagonal structure at high and the monoclinic or triclinic form at low temperature. This transition is known as the Brill transition [4]. It is widely accepted that PA6 does not reveal the full Brill transition. However our synchrotron wide angle x-ray diffraction (WAXD) experiment shows that the more rapidly cooled sample the more imperfect the  $\alpha$  phase and the more evident is the Brill transition upon heating. Surprisingly, the most clear Brill transition is evident upon heating of a sample annealed for 10 hours at 200°C, *i.e.* close to the melting temperature. This result, shown in Fig. 1, contradicts conventional wisdom that the  $\alpha$  form is the thermodynamically most stable, especially when formed during annealing at high temperatures.

We suggest that the  $\beta$  form of nylon 6 is of the same nature as the pseudo-hexagonal phase that occasionally appears during heating (Brill transition). Most likely  $\alpha$  crystals during heating convert into the transient mesophase of intermediate order (the  $\beta$  form). Increasing the temperature yields two opposing effects: On the one hand, thermal activation leads towards destruction of crystals and melting. On the other hand, thermal activation accelerates the transition to better ordered states via the  $\beta$  form. The outcome of the interplay of the two local processes depends on the morphology at hand and time allowed in the given temperature window.

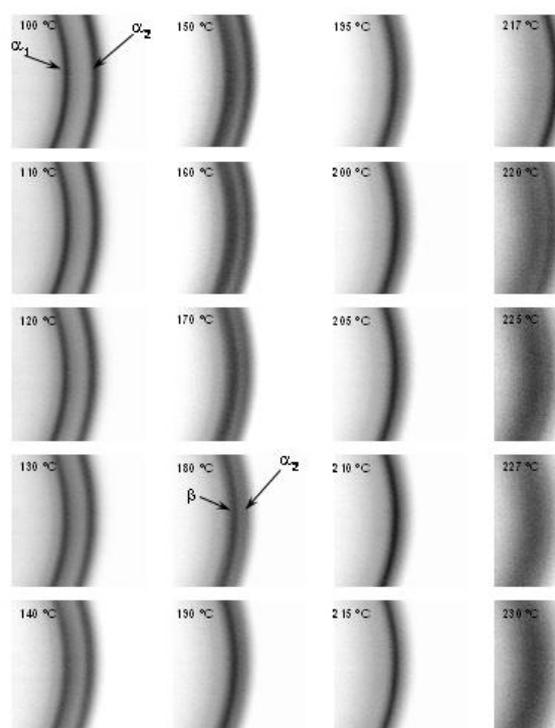


Figure 1. Selected 2D WAXD patterns of the annealed PA6 sample recorded on heating at 5°C/min.

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### References

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