

**Figure 13.4** Relationship between the tangent of the slope in temper curve at the inflection point  $(m_{_{\rm IP}})$  and the inflection point temperature  $(T_{_{\rm IP}})$ . Open symbols: seed tempered; closed symbols: conventionally tempered.

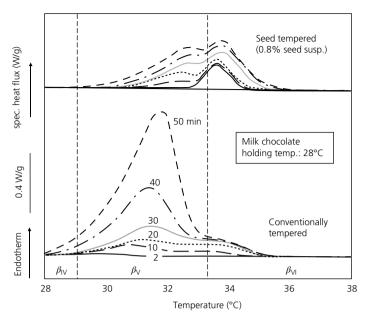
these conventionally tempered samples form a larger fraction of unstable polymorph crystals during supercooling than the seed tempered ones is because they contain more bigger crystals, and hence larger volumes of fat containing no seed crystals at all.

In addition the function  $T_{IP}(m_{IP})$  will vary according to the chocolate recipe (milk or plain), types of cocoa butter or other fats present and any emulsifiers that are used.

When comparing conventional tempering machines using the same recipe chocolate, a higher  $T_{\rm IP}$  value can be taken as an indicator for more stable crystals being present. Both the shape of the curve and the temperature of the inflection point should be recorded when taking cooling curve measurements and this is done by most of the commercial tempermeter instruments (see Chapter 24).

## 13.4.2 Differential scanning calorimetry

Differential scanning calorimetry (DSC) measures phase transition heat during cooling (crystallisation) or heating (melting) with controlled temperature/time gradient applied to a small sample volume (in order to provide near homogenous temperature conditions; see also Chapter 24). The so-called direct DSC method developed by Breitschuh and Windhab (1998) makes it possible to measure the crystal fraction and the polymorph distribution within a tempered chocolate masse. This method involves sampling in DSC aluminium pans for short sampling times ( $\leq 60 \, \text{s}$ ), under isothermal conditions [e.g.  $T \approx 28 \, ^{\circ}\text{C}$  (82  $^{\circ}\text{F}$ ) for a standard milk chocolate standard: 30  $^{\circ}\text{C}$  (86  $^{\circ}\text{F}$ ), dark chocolate] and with a constant sample weight ( $\approx 5 \, \text{mg}$ ). If these requirements are met, reproducible measurements of the seed crystal nuclei generated in the tempered chocolate system can be obtained.



**Figure 13.5** DSC measurements. Melting curves measured by the DSC direct method according to Breitschuh and Windhab (1998) for milk chocolate held at 28 °C (82 °F) under constant moderate stirring for different residence times.

Figure 13.5 shows DSC measurements for differently tempered (conventional and seed-tempered) milk chocolate held at 28 °C (82 °F) for different mean residence times. This simulates processing conditions for chocolate masses, for example being kept in the depositor of a moulding or enrobing machine. The different starting conditions for the conventionally and seed-tempered chocolate masses enable their respective total crystal fraction and polymorph distribution to be calculated. The development of the crystalline component is also shown as a function of residence time at 28 °C (82 °F; Figure 13.5). For the conventionally tempered milk chocolate there is a significantly faster increase in total crystal fraction with time and a clear dominance of the  $\beta_{\rm v}$  polymorph, whereas the  $\beta_{\rm vI}$ -seeded system increases in  $\beta_{\rm v}$  and  $\beta_{\rm vI}$  fraction in a more balanced manner, but with a clearly lower total crystal fraction.

## 13.4.3 Thermorheometry

Thermorheometry is a complementary technique for temper evaluation with the advantage of dynamic measuring conditions, which can be adjusted to come close to the conditions within a tempering machine. In general, the (apparent) viscosity is measured as a function of time for constant (apparent) shear rate. After distinct residence times under constant shear (e.g. at  $1\,\mathrm{s}^{-1}$ ) shear rate sweeps preferably between 0.1 and  $100\,\mathrm{s}^{-1}$  are carried out. Flow functions  $\tau(\gamma)$  for a milk chocolate masse shear treated at a shear rate of  $1\,\mathrm{s}^{-1}$  for  $3000\,\mathrm{s}$  ( $50\,\mathrm{min}$ )