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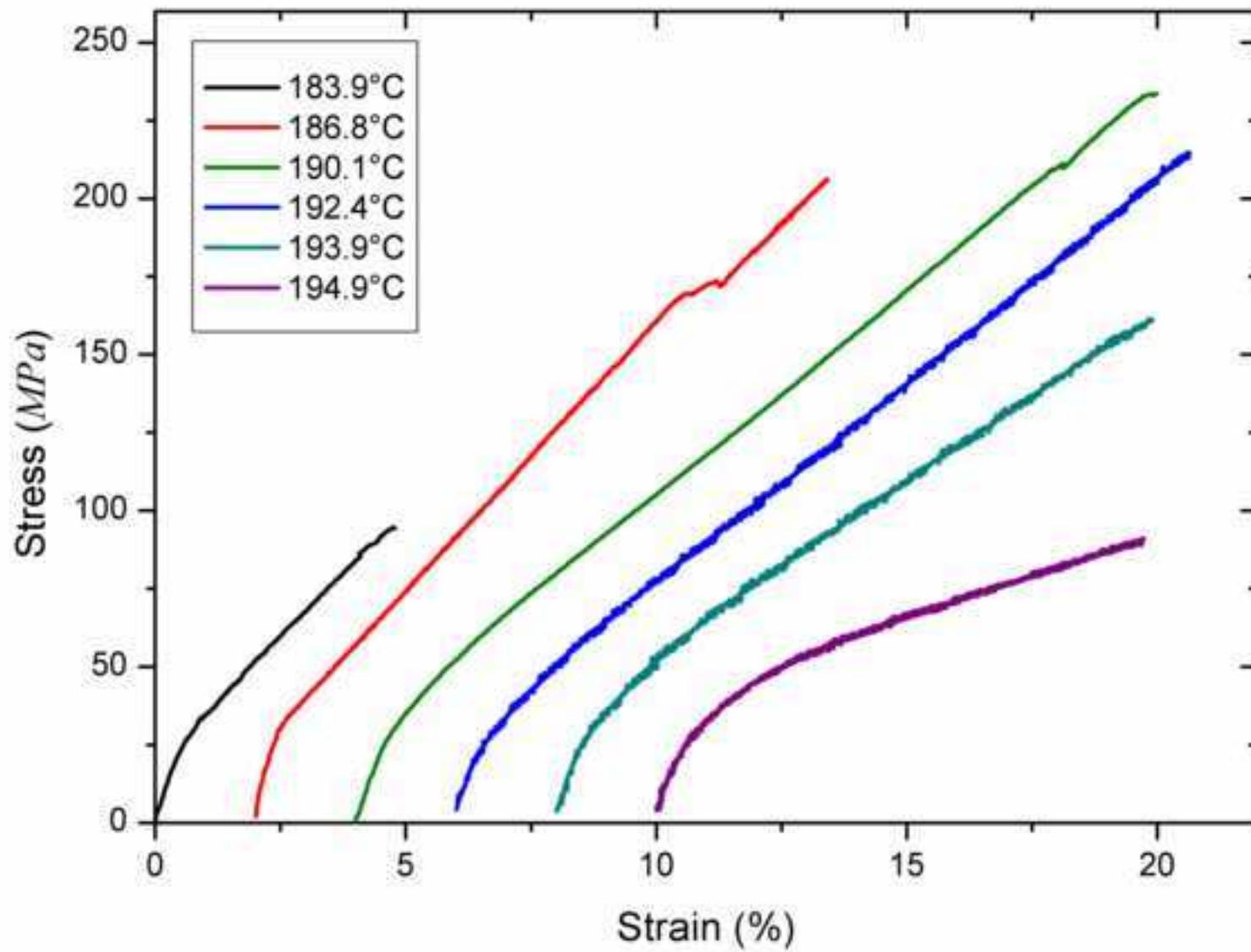
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## **The effect of draw ratio on the mechanical properties and crystalline structure of single polymer polypropylene composites**

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### **Abstract**

The properties of self-reinforced single polymer composites produced by the Leeds hot compaction process are highly dependent on the compaction temperature as well as the constituent oriented elements used to produce the compacted sheets. In this paper, the variation in tensile mechanical properties of uniaxial hot compacted sheets manufactured from drawn polypropylene (PP) tapes with change in compaction temperature have been investigated, for a range of different draw ratio tapes. It is shown that there is a measureable difference between the optimum compaction temperatures required for obtaining the highest modulus and strength in the compacted sheets. The compaction temperature required to achieve the maximum tensile modulus was seen to increase with increasing draw ratio. The compaction temperature to obtain the maximum tensile strength was found to be both independent of the draw ratio and a few degrees higher than that for obtaining the maximum modulus. Peak modulus and peak tensile strength was shown to be dependent on the draw ratio of the drawn tape.

Differential scanning calorimetry measurements on the compacted sheets were also performed in order to investigate the change in crystalline structure with compaction temperature and draw ratio. This has shown that the changes in structure within the oriented phase (i.e. tapes) during the compaction process itself are directly related to the final properties of the hot compacted sheets.

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### **1. Introduction**

There have been a number of different methods reported for the production of single polymer composites, from the first example, introduced by Capiati and Porter [1], using polyethylene filaments and a polyethylene powder with different melting points, to other techniques such as film stacking, powder and solvent impregnation [2-6], bi-component tapes with a lower melting point outer layer [7] and compression controlled melting [8]. All of these techniques require a matrix and reinforcing phase that, although of similar chemical composition, are different. A complete review of these techniques and other single polymer composites can be found in Ref. [9].

The hot compaction process, developed at the University of Leeds, has been applied to a number of oriented polymers for the production of single polymer composites [10, 11]. As distinct from the other processes described above, the hot compaction process uses only a single starting material. In this process, an assembly of oriented fibres or tapes (often in the form of a woven cloth) is taken, under a small constraining pressure, to a critical temperature where a thin skin on the surface of each oriented element melts. On cooling, this skin re-crystallises to form the matrix of a single polymer composite, with the remaining oriented fraction acting as the reinforcing phase. The virtue of this technique is that the matrix phase is produced around each fibre by melting and re-crystallisation, negating the need for a second phase. This gives excellent bonding between the remaining initial oriented phase and the melted and re-crystallised phase.

With all fibre reinforced composites, the properties of the final material are highly dependent on the properties of the original constituents and the fraction of each phase in the final material [12]. Typically most fibre reinforced composites have a maximum reinforcement phase volume fraction of 50-60%. In the hot compaction technique, the matrix phase is produced from the reinforcement fibres, and so a higher volume fraction of reinforcement is achievable. In previous work on polyethylene, analysis of the structure through a combination of microscopy and differential scanning calorimetry [10, 11] has shown that the optimum properties of the final composite were achieved at 30% of melting, i.e. 70% oriented phase remaining. Since the volume fraction is so much higher, this makes the original properties of the oriented phase even more critical. A further intriguing possibility, which is unique to these materials, is that the properties of the reinforcing phase can be changed by the processing stage. Taking a highly oriented polymer fibre or tape inside the melting range could result in morphological changes (such as re-crystallisation or loss of molecular orientation) which can be both temperature and time dependent.

A number of different studies have looked into the variation in mechanical properties with compaction temperature, in order to determine the optimum processing conditions: examples include polyethylene [10], polypropylene [13], PET [14] and Nylon [15]. A full review of the science and technology of hot compacted composites from a number of different polyolefins can be found in the review by Ward and Hine [16]. Differential scanning calorimetry (DSC) has proved a very useful technique for examining morphological changes over the melting range of each particular oriented fibre or tape, since the enthalpy of melting is a direct measure of the total crystallinity of the composite.

In this study, we report the change in mechanical properties and the link to morphology of hot compacted, single polymer polypropylene composites produced from oriented tapes with a range of draw ratios. The effects of detailed, precise changes of the hot compaction temperature, over a range around the optimum compaction temperature, and the effect of draw ratio on the properties of the resultant composite sheets have been investigated, looking at the crystalline structure of the composites changing during the hot compaction itself using DSC analysis.

## **2. Experimental**

### *2.1. Materials*

The polymer used in this work was a polypropylene (PP) homopolymer with a weight average molecular weight ( $M_w$ ) of 360,000 g/mol, a number average molecular weight ( $M_n$ ) of 78,100 g/mol, a density of 910 kg m<sup>-3</sup>, and an unconstrained peak melting point of 163 °C.

The material was melt-spun into tapes using a Betol 2525J single screw extruder with a slot die (10x0.2mm). A temperature profile of 175, 200, 215 and 225 °C was used for the four heating zones along the barrel, and the heating zones in the die were set to 235 and 240 °C respectively. The screw speed was set to ~5rpm. The extruded tape was passed onto heated rollers (100 °C and ~12rpm) and hauled-off under a slight tension. The melt-spun tapes were drawn to four different draw ratios ( $\lambda$ ), of 6.2±0.3, 10.0±0.6, 13±1 and 14±1, through a hot air oven, at a drawing temperature of 170 °C in a continuous process.

### *2.2. Hot compaction*

All the hot compacted samples produced in this study were unidirectional samples (55mm x 55mm and 0.5 mm thick), i.e. the tapes were aligned in a single direction. Samples were made by winding the tapes around a metal picture frame, which was then placed under a pressure of 4.9MPa in a hot press and taken to the compaction temperature for a set dwell time (5 min) before fast cooling at a rate of around 50 °C/min. Samples were made at a range of compaction temperatures and the compaction temperature itself was determined as the peak temperature reached during the compaction process, monitored by means of a thermocouple embedded within the compacted sheet, along the edge of the sample area.

### *2.3. Mechanical Testing*

The Young's modulus, maximum stress and strain at maximum stress of the hot compacted sheets were determined by static tensile testing on an RDP-Howden servo-mechanical tensile testing machine. Dumbbell shaped samples were cut from the hot compacted sheets, consistent with ASTM D638, along the longitudinal direction. Samples were tested at 21 °C, 50%RH, and a nominal strain rate of 5x10<sup>-3</sup>s<sup>-1</sup>.

Sample strain in the tensile tests was determined using a Messphysik video extensometer to track targets painted onto the sample surface. To avoid sample straightening affecting the measurement of Young's modulus, a pre-load of approximately 1.5MPa was applied.

### *2.4. Differential scanning calorimetry*

DSC measurements were carried out using a Perkin Elmer Series DSC-7 in order to determine morphology changes during the hot compaction process. Scans were run from 120 °C to 220 °C at a rate of 10 °C per minute. The total enthalpy of the hot compacted samples was determined from the area under the DSC melting peak, as defined by the deviation from an extrapolated baseline, with fixed endpoints at 140 and 185 °C.

During the hot compaction process, the polypropylene sample undergoes a combination of melting of the skin of the oriented tapes into an isotropic melted phase (which binds the compacted structure together), and possible rearrangement or annealing of the internal crystalline structure of the tapes. The hot compaction process therefore changes the oriented polypropylene sample morphology depending on the compaction temperature, in addition to producing the required amount of selectively melted matrix material to bind the structure together. By examining the change in enthalpy (which is proportional to the crystallinity [17, 18]) of the hot compacted samples at different compaction temperatures and draw ratios ( $\lambda$ ), it is possible to probe the morphological structure changes with compaction temperature and assess how that changes with the different draw ratio tapes used to make the compacted sheets.

## **3. Results and discussion**

### *3.1. Mechanical properties*

Figure 1 shows typical stress-strain curves for a series of the 10 $\lambda$  hot compacted samples produced at a range of compaction temperatures, between 184 and 195 °C. The variation in mechanical properties with compaction temperature can quite clearly be seen, in terms of Young's modulus, maximum stress and strain to failure (the samples have been offset along the strain axis by 2% to allow the behaviour to be more easily viewed). At the lowest compaction temperature, it is proposed that there is insufficient melted and recrystallised material to produce a homogeneous well bonded structure. As the temperature increases the single polymer composite properties first improve as the fraction of selectively melted material increases and then fall as substantial crystalline melting occurs. Clearest in this representation is the peak in tensile strength at a compaction temperature of around 190 °C.

The variation in the Young's modulus with compaction temperature for the four draw ratios is shown in Figure 2. It is immediately apparent that the Young's modulus of the samples for each draw ratio goes through a peak at a particular compaction temperature. The values are 186, 187, 188 and 188 °C for the 6, 10, 13 and 14 $\lambda$  samples respectively. This peak in modulus is obviously of interest since this represents the optimum tensile stiffness of the compacted sheets. The compaction temperature corresponding to this peak is defined as the optimum compaction temperature for each draw ratio, for this particular mechanical property.

The peak in Young's modulus with increasing compaction temperature is expected, and has been seen in previous work on hot compaction [10, 13, 19-21]. This occurs because the hot compaction process is a competition between selective surface melting of the oriented phase, with loss of molecular orientation at the same time. The essence of the hot compaction process is that it involves the melting of the oriented elements in order to provide the matrix material needed to bind the structure together and produce the composite sheet, with the melted material recrystallising on the surface of the tapes during cooling. The key is to produce sufficient matrix material without losing the molecular orientation of the oriented tapes. The results suggest that as the draw ratio is increased, the thermal stability of the oriented structure is increased and hence the optimum temperature also increases (in order to create the same amount of melted matrix material).

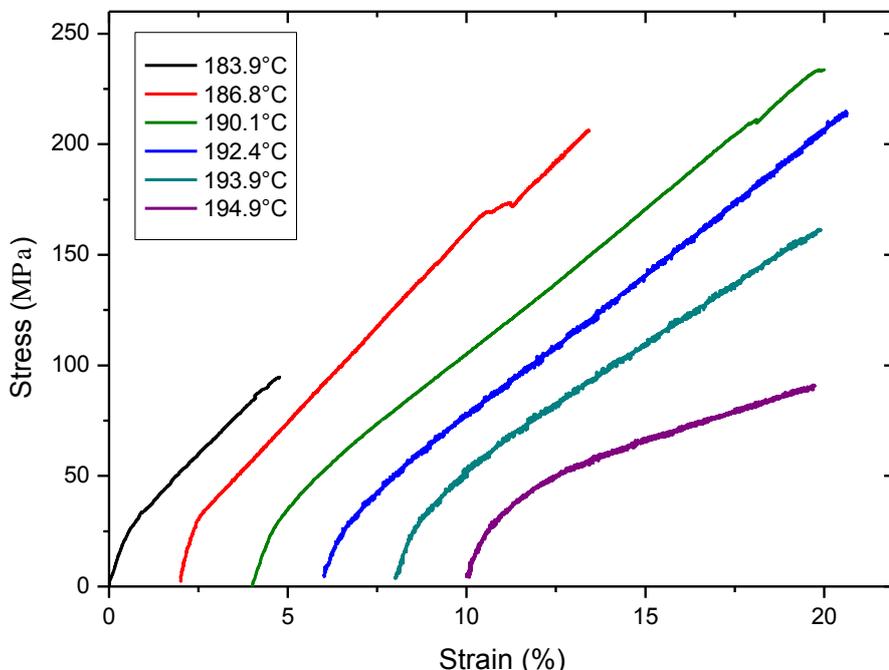


Figure 1 Typical stress-strain curves for hot compacted samples produced at different compaction temperatures. Note that the curves have been shifted horizontally along the strain axis for display purposes.

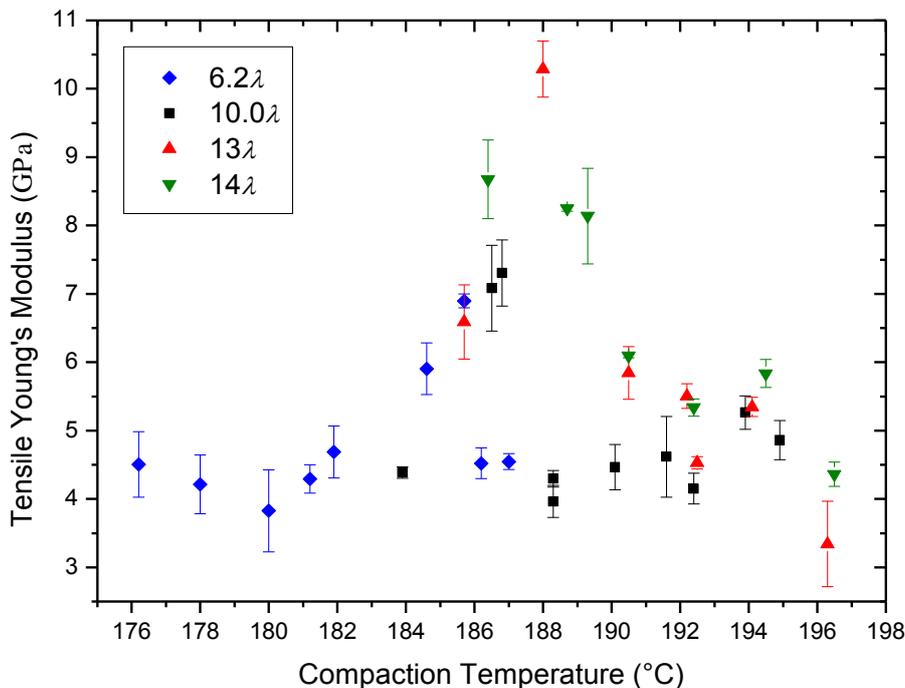


Figure 2 Variation in tensile Young's modulus with compaction temperature of hot compacted samples produced from the four different draw ratio tapes in this study. A noticeable peak in modulus is seen for all four draw ratios.

As the compaction temperature is increased, there is a greater amount of melting along the surface of the drawn tapes, and so more material is available in order to bind the structure together. The observed increase in Young's modulus with compaction temperature is thus due to the fact that at low compaction temperatures, there is not enough melted material to bind the composite sheet together effectively, and so failure occurs along the interface of the tapes, resulting in the low measured Young's modulus. As the compaction temperature is increased, the amount of material increases, up to the point where the structure is fully bound together, resulting in the optimum stiffness of the compacted sheets. This has been confirmed in previous morphological investigations on compacted materials [10, 11, 22, 23]. At higher compaction temperatures, the Young's modulus decreases, because greater amounts of the remaining oriented fibre core from the original drawn tapes is lost to the matrix material, and so the overall composite stiffness is reduced.

Figure 2 also confirms that the overall Young's modulus of the compacted sheets at the optimum compaction temperatures increases with the draw ratio of the original drawn tapes, up to a draw ratio of  $13\lambda$ . The  $14\lambda$  samples have a lower peak modulus because of structural changes to the tapes during the compaction procedure. Although the tapes are constrained around the metal frame during the process, molecular relaxation appears to be occurring at the highest draw ratio that could be achieved. This suggests that the draw ratio of 13 is optimal for these single drawn tapes.

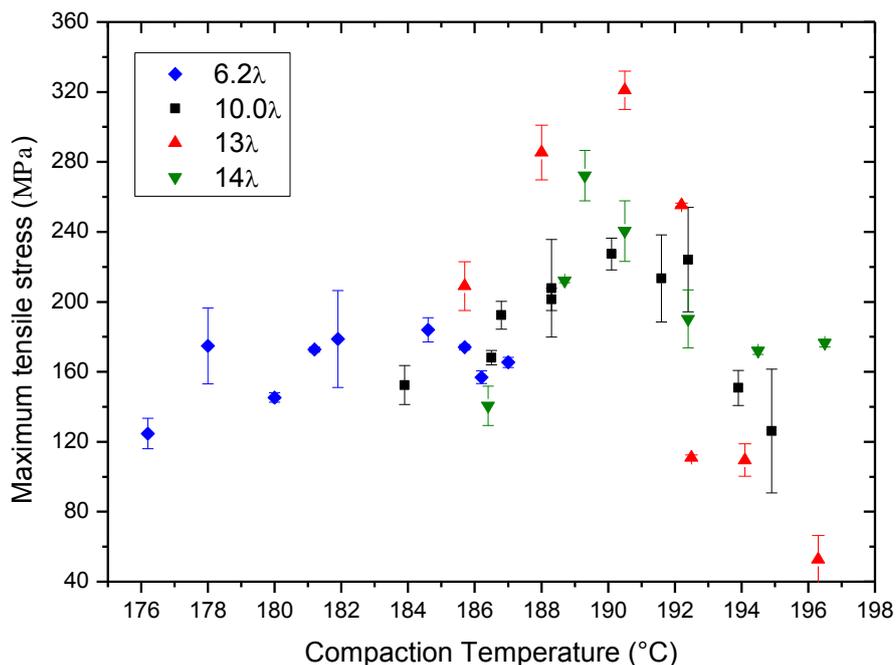


Figure 3 Maximum (failure) stress of the hot compacted samples against compaction temperature for all samples produced from the four different draw ratio tapes. Note that the peak in the maximum stress is at a higher compaction temperature than that seen for Young's modulus in Figure 2.

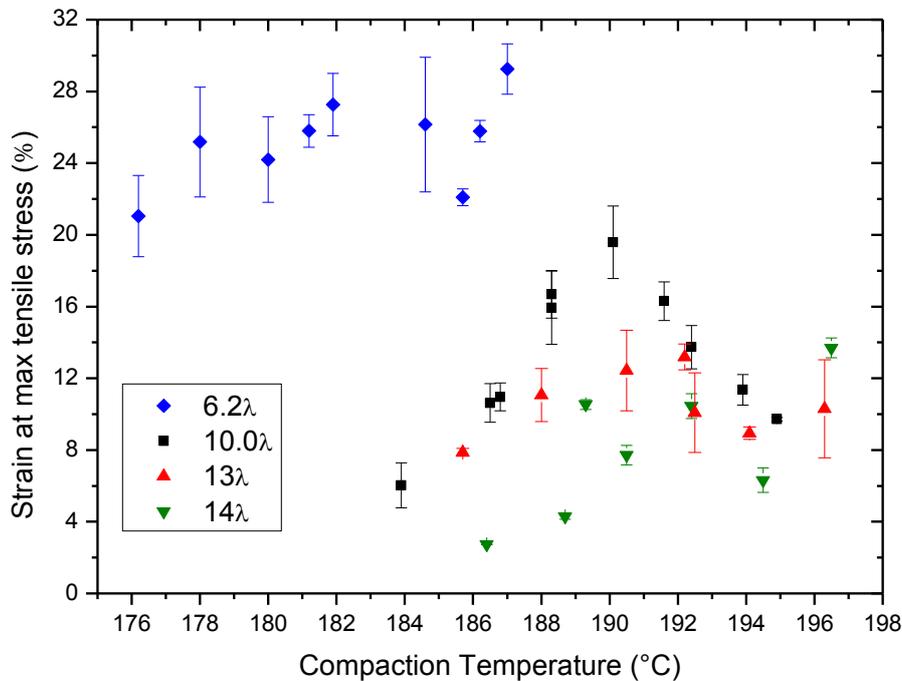


Figure 4 Strain at maximum stress of the hot compacted samples against compaction temperature for all samples produced from the four different draw ratio tapes. The peak strain at max stress occurs at the sample compaction temperature as the max stress, as seen in Figure 3.

Figure 3 shows the maximum (or failure) stress with compaction temperature and Figure 4 shows the strain at this maximum stress against compaction temperature. As with the Young's modulus, the failure stress values also go through a peak at a particular compaction temperature. It is important to note that this peak occurs at a higher compaction temperature than the optimum compaction temperature, as defined by the peak in Young's modulus, that the peak in properties is much broader than that seen for the tensile modulus, and finally that the peak in properties occurs at a similar temperature for all draw ratios (~190°C), excluding the lowest draw ratio of 6.

An important new finding is also that the peak in maximum tensile stress and peak in strain at maximum tensile stress occur at the same compaction temperature, because both the maximum stress and strain are determined by the same temperature.

The peak in failure of the compacted sheets occurs because the method of failure varies depending on the compaction temperature at which the samples are produced. At lower compaction temperatures (below optimum), failure is almost exclusively due to poor bonding between the tapes since there is an insufficient amount of matrix material to bind the structure together. Above the optimum compaction temperature, the strength of the sample reaches a broad peak and then decreases again at higher compaction temperatures (about 3-4°C above the optimum), due to widespread crystalline melting and loss of orientation. While the modulus is very dependent on the level of local molecular orientation and local crystalline structure, the failure strength is usually considered more dependent on molecular orientation at the length scale of the polymer chain, or more generally, molecular weight. As the compaction temperature is increased through the melting range, molecular re-organisation first occurs at a local scale, leading to a potential loss of modulus. However, preferred orientation at the length scale of the polymer chain can be retained for a longer time, due to the much greater relaxation time of this property this has been shown in a recent paper for amorphous polystyrene [24].

This broader compaction window for achieving maximum strength is important for some commercial applications for the technology. One reason is because achieving maximum Young's modulus in the hot compacted material requires careful control of the temperature during the compaction process due to the narrow compaction window, and so a broader window (and higher temperature) is a significant advantage.

The variation in the peak of the maximum stress of the hot compacted sheets with draw ratio follows the same trend as that seen in tensile Young's modulus, the peak maximum stress increasing with draw ratio, with the 13λ samples showing the highest peak max stress. The 14λ samples showed a reduction in peak maximum stress, for the same reason as discussed above.

The strain at peak maximum stress predictably shows a significant decrease with increasing draw ratio, since the method of hot drawing the original tapes to higher draw ratios extends and aligns the polymer chains, and so reduces the ductility in the final composite. By normalising the properties of the compacted sheets to the original tape properties (i.e. by dividing the composite property by the same property of the original drawn tape), it has been shown that the 13λ samples result in the best overall translation of mechanical properties from the drawn tapes [25], taking into account both stiffness and strength.

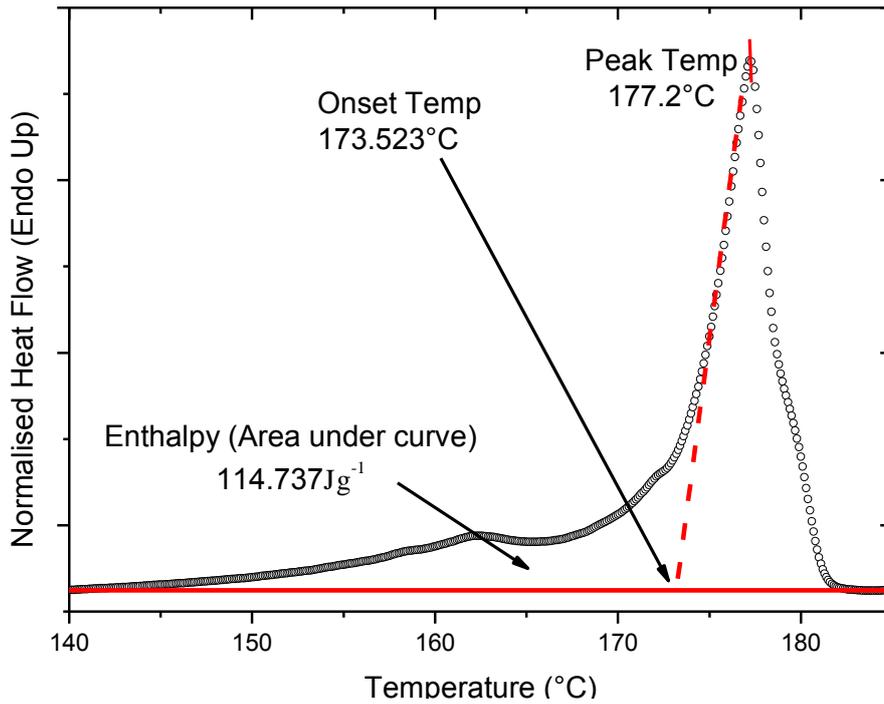


Figure 5 DSC melting endotherm for a hot compacted sample produced at a hot compaction temperature of 191.6°C. This sample was produced from 10λ drawn tapes. The enthalpy of melting (area under the curve) was determined by using fixed points to determine the baseline.

Figure 5 shows a typical DSC melting endotherm for a sample compacted at 191.6°C produced from 10λ drawn tapes. The enthalpy of melting for the sample was determined from the total area underneath the melting peak, using fixed points to define the baseline for all samples.

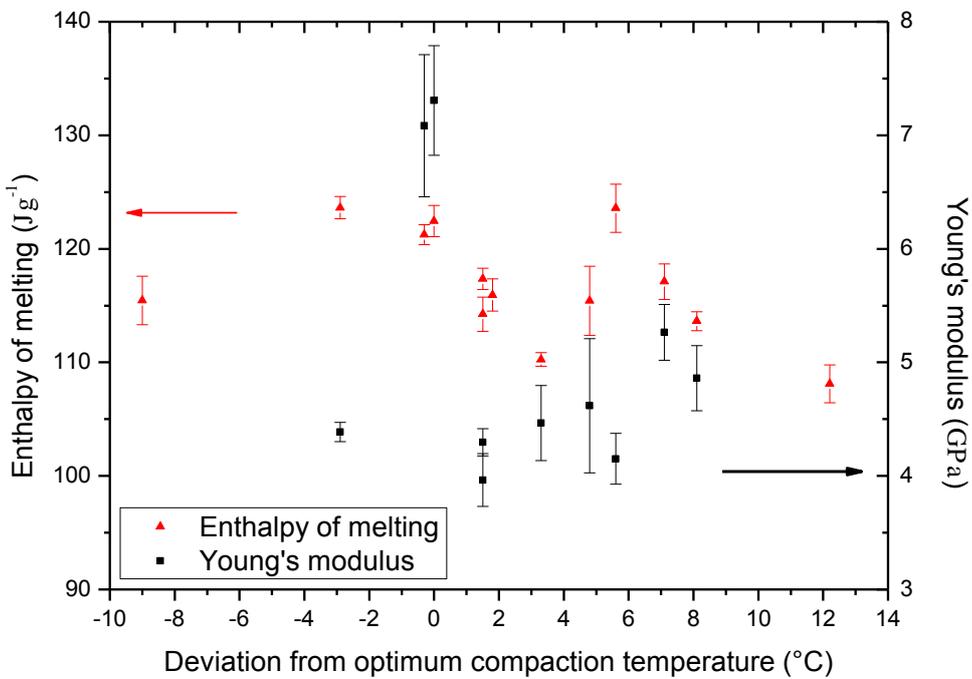


Figure 6 Enthalpy of melting (▲) and tensile Young's modulus (■) against compaction temperature (as a deviation from the defined optimum compaction temperature) for 10λ compacted samples.

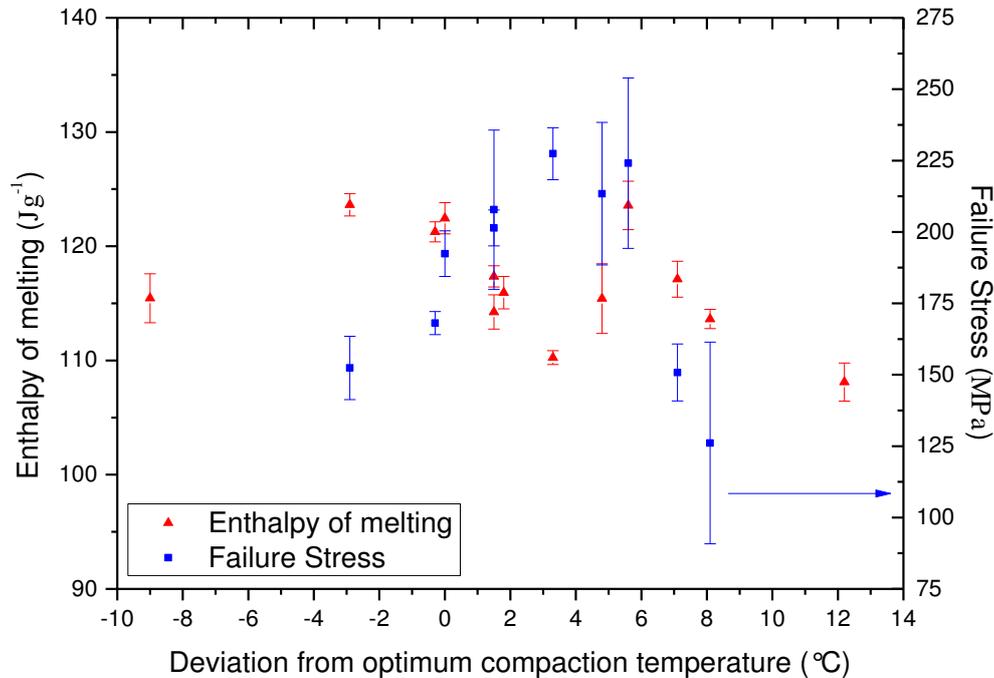


Figure 7 Enthalpy of melting ( $\blacktriangle$ ) and maximum (failure) stress ( $\blacksquare$ ) against compaction temperature (as a deviation from the defined optimum compaction temperature) for  $10\lambda$  compacted samples.

The total enthalpy of melting is plotted in Figure 6 as a function of the compaction temperature. Normalising this data for each draw ratio and plotting on the same figure showed that the distance between the two peaks in enthalpy decreased with increasing draw ratio.

This dependence showed an increase in total enthalpy up to 1-2°C below the optimum compaction temperature for the modulus, at which point the enthalpy shows a sharp decrease to a minimum value 2-3°C above the optimum compaction temperature. This is then followed by a much sharper 2<sup>nd</sup> peak in enthalpy, approximately 6°C above the optimum compaction temperature, before decreasing again. This trend was most clearly shown by the  $10\lambda$  samples produced and these results are shown in Figure 6 and Figure 7.

Figure 6 shows the comparison between the variation in total enthalpy with compaction temperature (normalised to the optimum compaction temperature for modulus), and the variation in Young's modulus of the samples. Figure 7 shows a similar comparison, between the same total enthalpy and variation in maximum tensile stress of the samples.

These results show the link between the structure of the hot compacted sheets at a particular compaction temperature and the two different mechanical properties. The optimum Young's modulus occurs just after the first peak in enthalpy which is when the structure is still highly ordered, because so much material remains in the oriented core of the tapes. Then local re-organisation of the crystalline structure begins to occur, initially leading to a reduction in crystallinity (and modulus) and then as the temperature increases, the structure recrystallises again, leading to an increase in enthalpy but not local orientation. However, molecular orientation at the length scale of the polymer chain is not yet affected and so the strength is maintained. Finally, as the compaction temperature is further raised, all the crystalline structure is lost and then strength finally falls. In conclusion, the fall in modulus is associated with the first fall in enthalpy and is associated with local crystalline reorganisation, which then can recrystallise leading to an associated increase in enthalpy. The strength is not so dependent on the level of local molecular orientation and therefore only falls when the final large scale melting occurs. This compares to previous studies on hot compacted polyethylene composites, which is highly crystalline and highly oriented, and thus shows no internal melting peak. The difference in structure, in comparison to the PP composites studied here, therefore resulted in the composite modulus and strength falling off at the same time with increasing compaction temperature [10].

#### 4. Conclusions

The production of hot compacted polypropylene sheets from a number of different draw ratio tapes has resulted in variety of interesting properties that have not been observed previously.

Measurement of the mechanical properties of the hot compacted sheets indicated that the Young's modulus, maximum stress and strain at the maximum stress all go through a peak with increasing compaction temperature. The optimum compaction temperature (peak in Young's modulus) was seen to be lower than that for the peak values for maximum stress and strain at maximum stress. The compaction temperature for peak maximum stress and peak strain at maximum stress was observed to be the same. These trends were seen for all draw ratios. It was also seen that the "compaction window" for obtaining maximum stress of the sheets was wider than that for obtaining optimum Young's modulus.

Differential scanning calorimetry indicates a change in the structure of the PP hot compacted sheets with increasing compaction temperature. There are strong links between the structural changes observed by DSC and the mechanical properties, particularly the maximum stress, of the final compacted sheet.

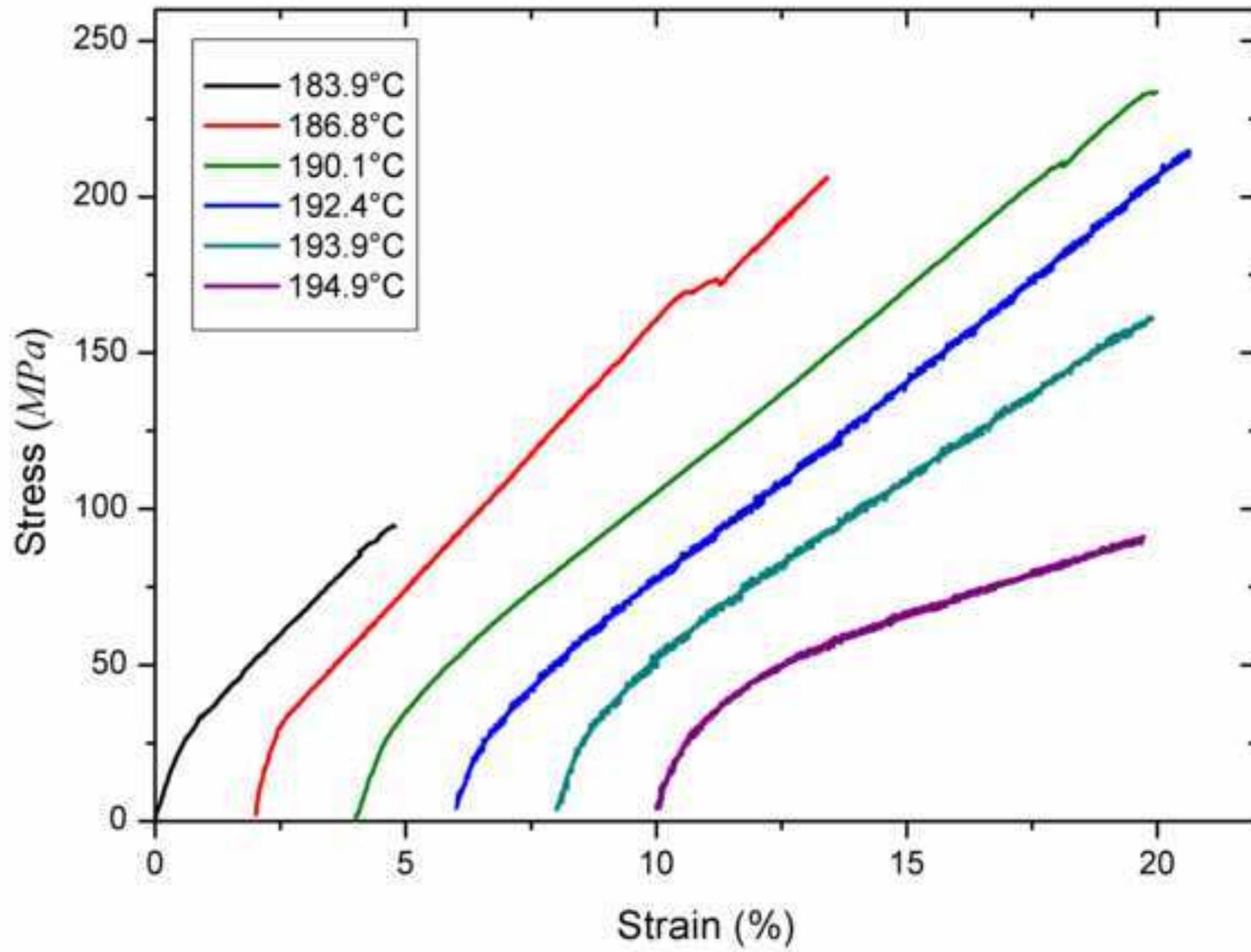
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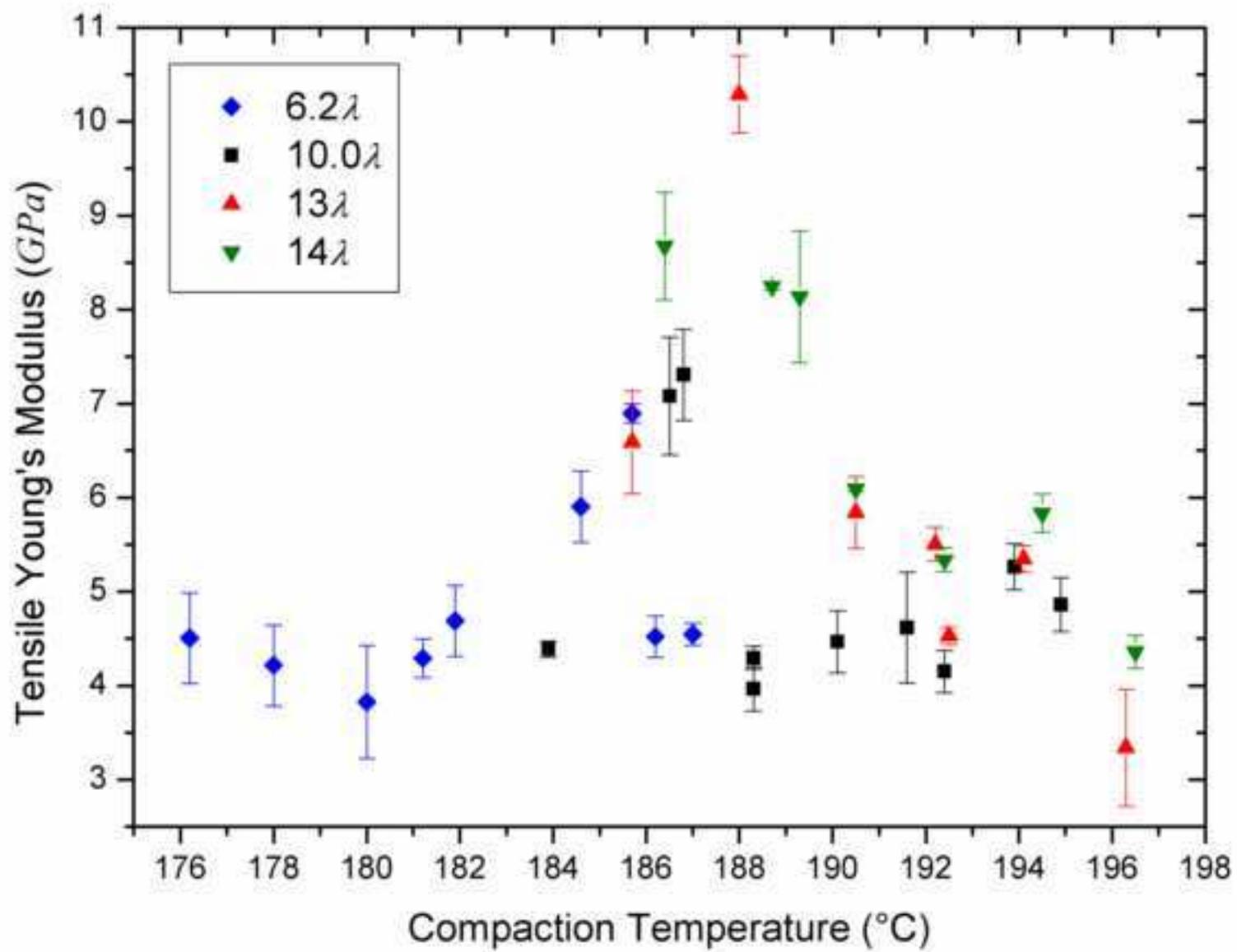
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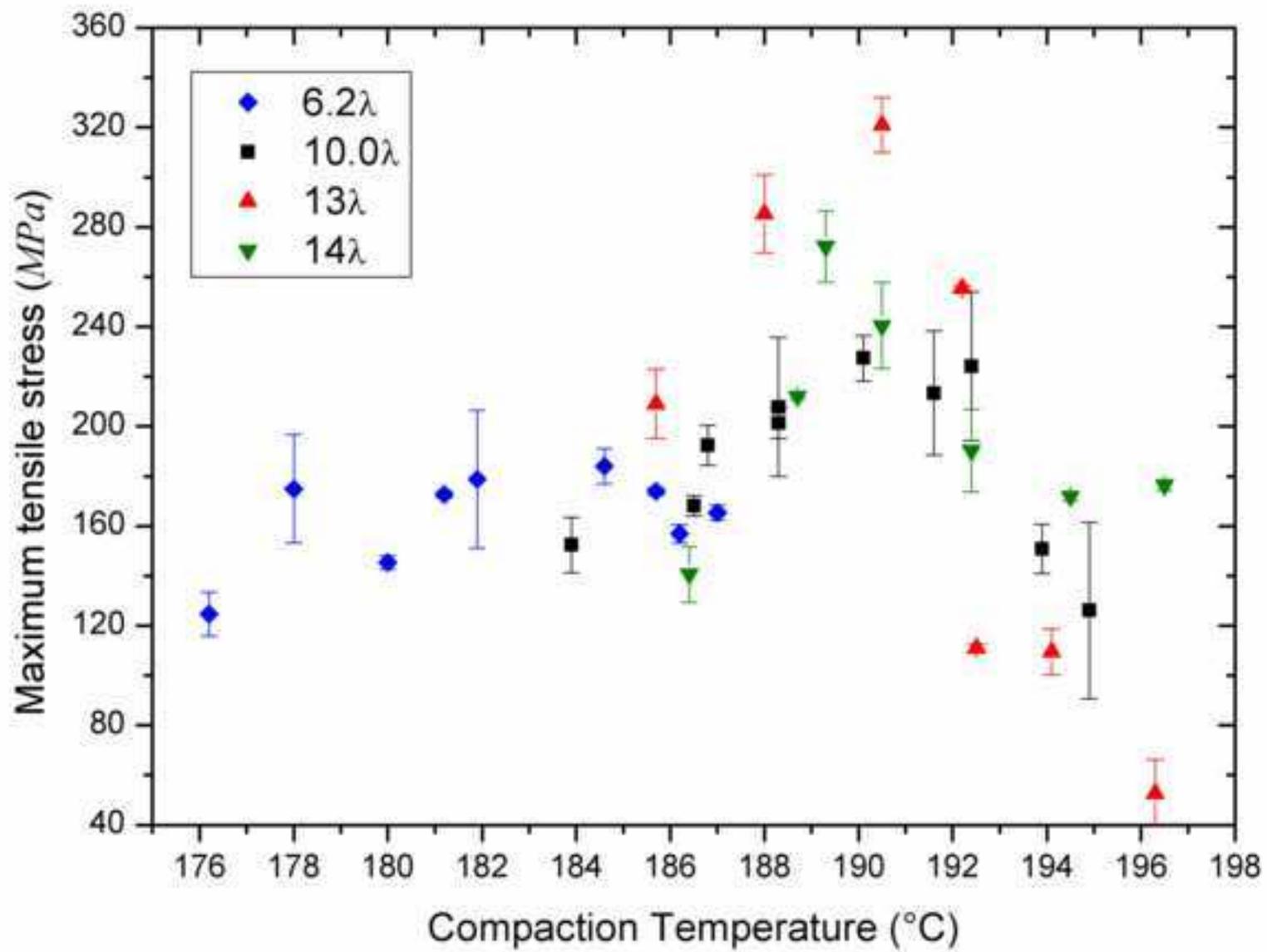
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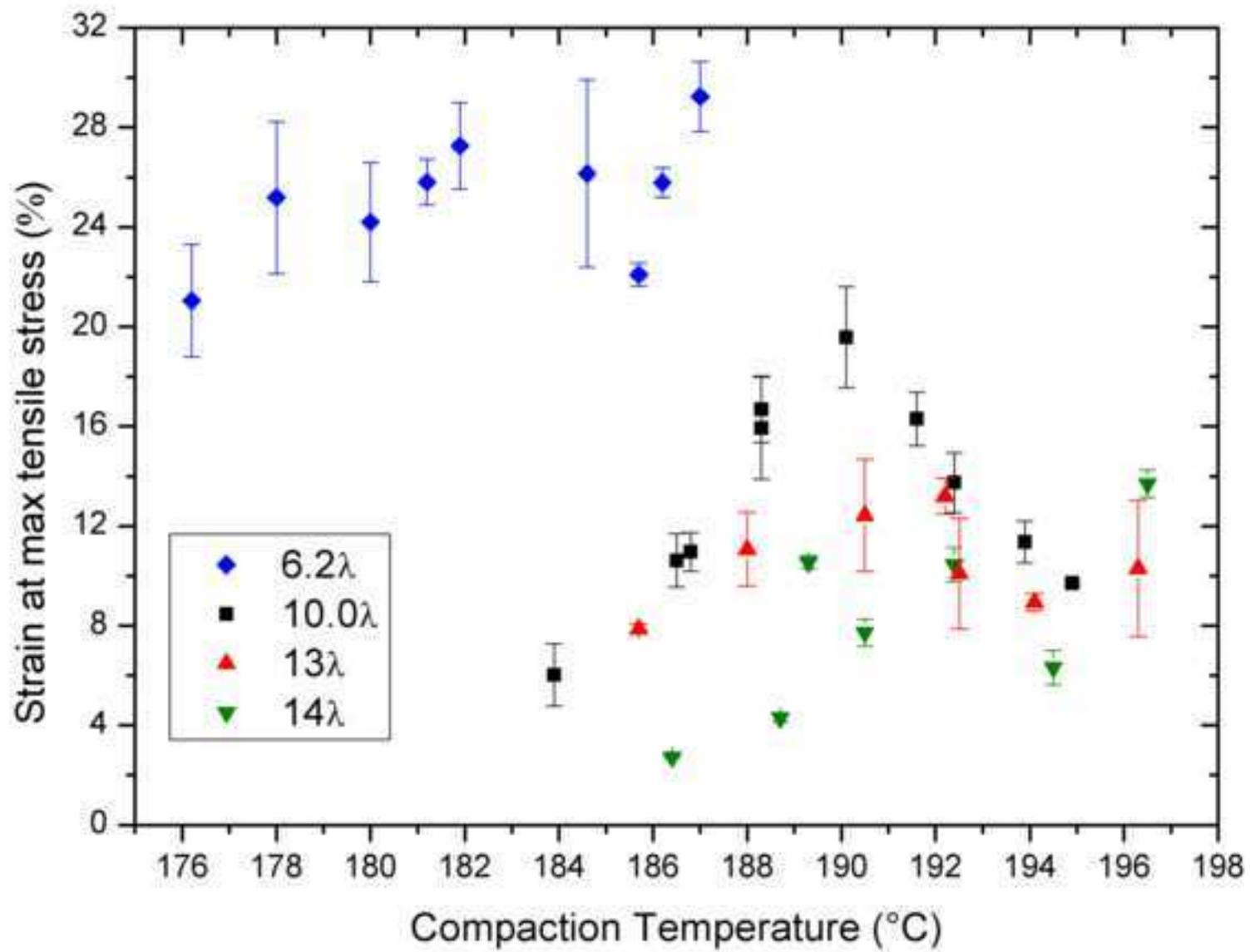
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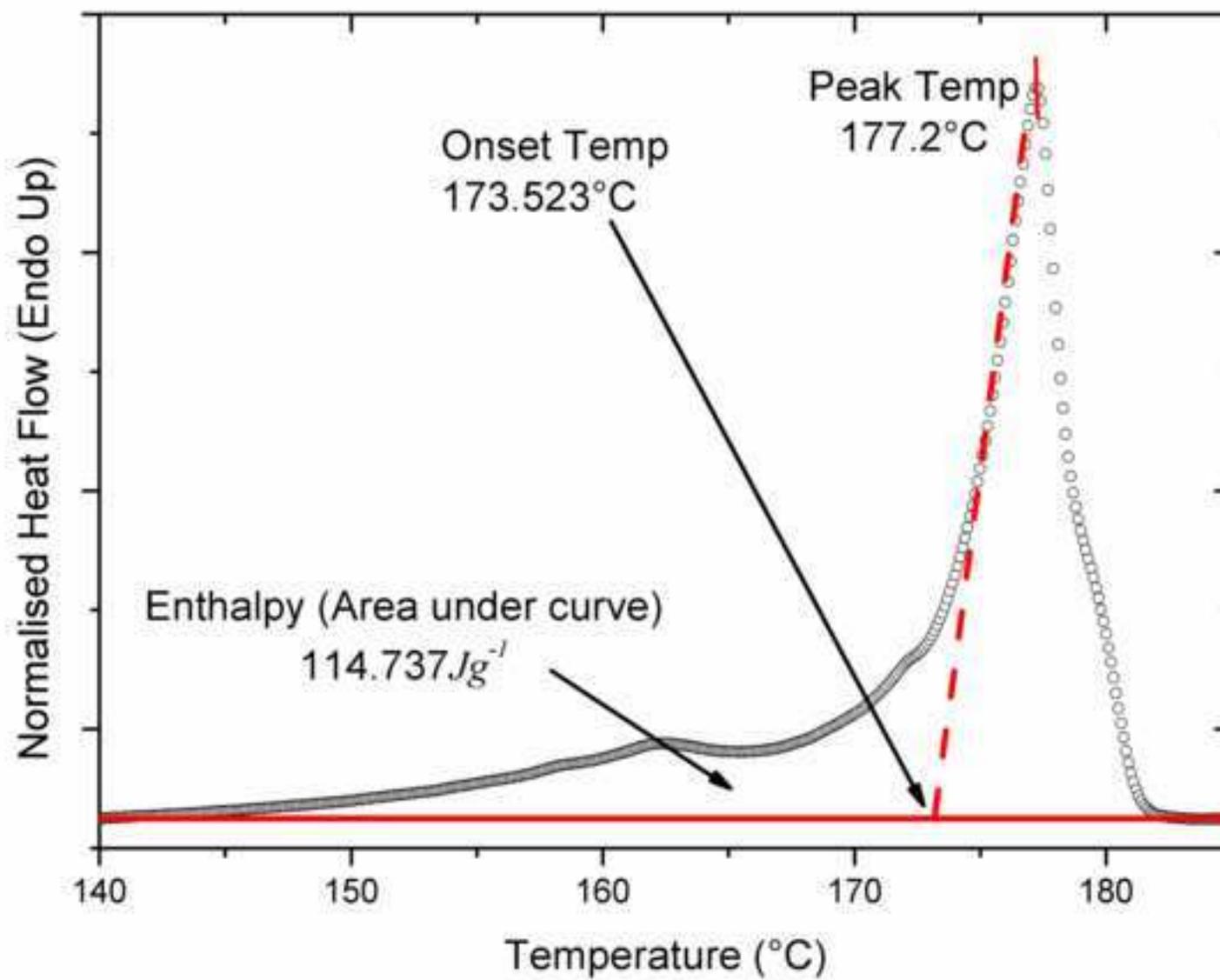


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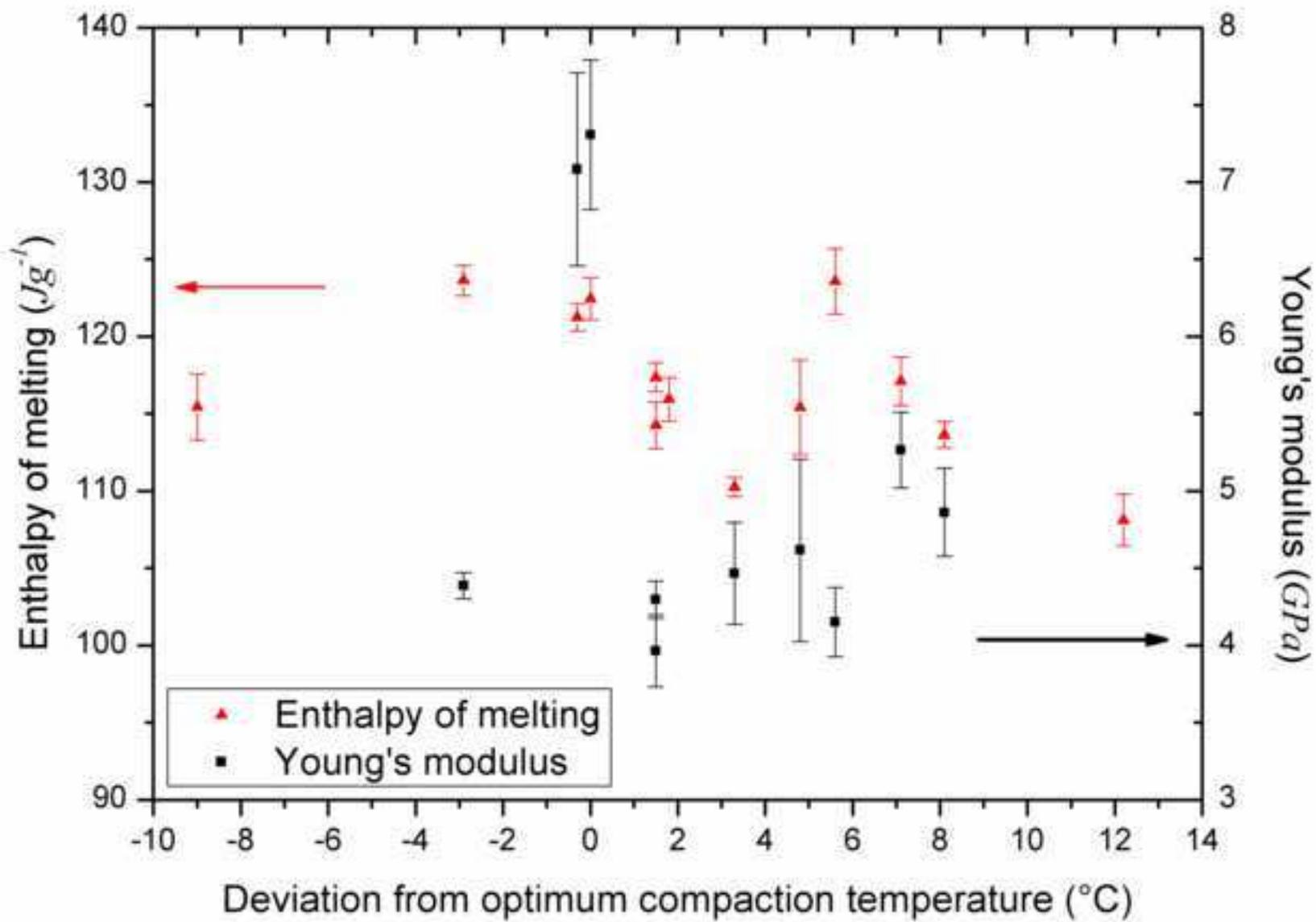


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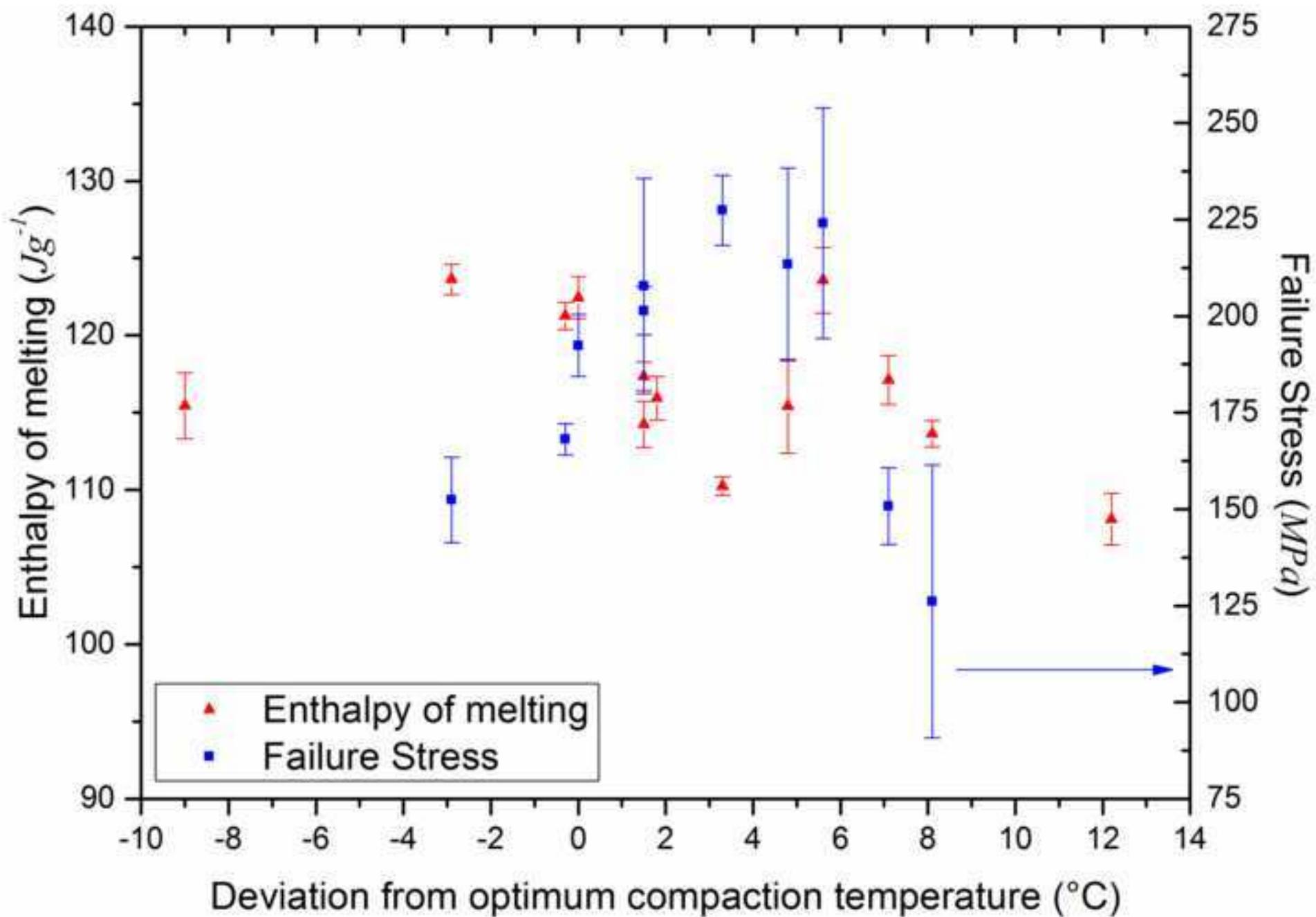
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**The effect of draw ratio on the mechanical properties and crystalline structure of single polymer polypropylene composites**

**Highlights**

- Max Young's modulus and failure strength require different processing conditions
- Crystalline structure change is directly related to change in mechanical properties
- Maximum failure strength is independent of draw ratio