APPLICATION NOTE

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Structural Changes in Polymers by Means of Cryogenic Grinding

(with kind permission of Prof. Dr. U. Köster, Dept. of Chemical Engineering, University Dortmund.)

One goal in materials research is to develop new synthetic materials with characteristics that cannot be accomplished by the usual smelting process, as the polymers tend to separate during the heating process.

Pure Polymer

Trials to introduce permanent changes in polymers by means of Cryogenic Mechanical Milling (CMM) with the Freezer/Mill® were started with two polymers; isotactic polypropylene (iPP) and syndiotactic polystyrol (sPS). After melting and re-crystallization, both cryogenically ground (i.e.at–196°C) polymers showed characteristics that differ from the original polymers. DSC (Differential Scanning Calorimetry) tests showed changed crystallization temperatures, which indicates reduced molecular weight or a change in molecular weight distribution. The longer the samples were subjected to cryogenic grinding the bigger the change.

Further experiments showed that the crystalline structure is increasingly destroyed during cryogenic grinding. Presumably CMM leads to chain fission and therefore to the formation of macro radicals. This can lead to cross-linking and an increased amorphic content in the partially crystallized polymers. Another explanation would be the formation of nano structures, small fragments of the original polymer compounds.

Compounds

As well as the pure polymer results the behaviour of cryogenically ground polymer compounds is, of course, very interesting. When mixing the two polymers iPP and sPS through melting (extrusion) separation occurs. Such polymer blends are dispersions of one part within the other. Some CMM sample tests suggest that re-combination of constitutive different macro radicals partly results in co-polymers. These co-polymers could act as phasing facilitators between the incompatible blending components of iPP and sPS. Evidence of this is the altered morphology of CMM blends. Here the DSC experiments also showed changed crystallization behaviour. More graphic are the morphology light microscopy pictures. While, after melting and re-crystallization, the extruded samples show morphology characteristics for roughly separated polymer blends, in a CMM sample the iPP is finely distributed within the sPS. (See Fig.)

Conclusion

CMM using the Freezer/Mill® causes changes to structure, thermal behaviour and morphology of homopolymers and polymer compounds. While morphological differences through melting and re-crystallization are partly reversible, structural changes remain. Aside of chain fission and the resulting macro radicals, cross-linking and reduced crystallization level has been observed. Polymer blends produced by CMM show a considerably improved dispersion of both polymers within each other. Exactly this changed degree of dispersion gives reason to hope for new materials with new and perhaps even predictable characteristics.

Application Note SP008:
Structural Changes in Polymers

Apparatus:
Freezer/Mill®

Application:
Polymer Blending

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Fig. A. Morphology of sPS/iPP (50/50ww) melting blends.

Fig. B. Of sPS/iPP (50/50 w/w) after cryogenic grinding; both re-crystallized after melting.