



Effect of magnetic field during the liquid – solid phase transition of some thermoplastic polymers

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ABSTRACT

Application of external fields on a polymer is common physical treatment for modifying the polymer properties. The field is applied on the final polymeric object or during synthesising or polymerization. In this work, the magnetic field from powerful permanent magnets was applied during the phase transition from liquid to solid phase, where the polymer chain inter-attraction forces are minimal. So, magnetic fields with less strength may perform valuable structural variations. It is found that magnetic field of 355 mT accelerated the spherulite growth in polyethylene glycol (PEG) films. High density polyethylene (HDPE) of zigzag chain, polypropylene (PP) of chains with side group and polystyrene (PS) as glassy polymer with ring, were magnetically treated with different field strength during their phase transition. Treated and untreated film samples were tested physically. IR- spectroscopy, X-ray diffraction, DTA, DSC and tensile stress – strain testing techniques were used in order to characterize, quantitatively, most types of the occurred variations if any. PS samples were casted by hot pressing and from dilute solution in magnetic field in order to detect the effect of solidification time on the size of the magnetic induced variations. Properties of treated HDPE showed little swinging about the untreated value in the field strength up to 160 mT. PP properties showed serious variations in the same range of field strength. PS films casted from dilute solution showed stronger variations than the films casted by hot pressing of the melted PS. All data are presented in coloured graphs. Spherulites images are extracted from a video clip that is used in measuring the rate of PEG spherulite growth in magnetic fields.

Indexing terms/Keywords

polymer phase transition; magnetic effects; spherulite growth; PEG; HDPE; PP; PS; polymer physical properties.



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Introduction

All substances have strong or weak magnetic property and have different responds to external magnetic field. Research on the effect of external magnetic field to polymer materials was originated from the discovery of molecule orientation arrange of liquid crystal molecules in magnetic field [1-3]. Molchanov et al. [4] firstly found the change in the supra-molecular structure of epoxy resin under the magnetic field in 1973. Since then, a series of experiments indicate that the curing reaction speed, glass transition temperature and thermal resistance are improved after applying magnetic field, and the mechanical properties of epoxy resin and its composites substantially increase as well [5, 6].

Polymers synthesised in the presence of a magnetic field often exhibit different properties and polymerisation kinetics when compared to polymers synthesised by traditional procedures [7]. Even weak magnetic fields, influence chemical kinetics and changes the activation energy and entropy for a chemical reaction, especially for polar compounds with high diamagnetic susceptibility [8, 9]. Other magnetic field effects include magnetic orientation and concentration, e.g. aligning the molecules and polymer chains. Polymers obtained within a magnetic field present enhanced thermal stability and lower swelling due to their ordered structure [7,8].

The origin of the diamagnetism is the induced magnetization caused by the induced motion of electrons under the applied magnetic field [10]. The magnitude of the magnetization M induced on a material is proportional to the applied field strength B , i.e., $M = \chi \cdot B$, where χ is the diamagnetic susceptibility. The magnitude of χ is typically in the order of -10^{-6} to -10^{-5} . Incidentally, this induced magnetization is also the source causing the chemical shift in the nuclear magnetic resonance (NMR) measurement. The interaction of M with the applied field causes a repulsive force, repelling the particle away to the direction of decreasing field strength. In the case that a material has anisotropy in diamagnetism, a magnetic torque acts on it, resulting in rotation. The origin of the magnetic anisotropy is traced back to chemical bonds. For example, diamagnetic susceptibilities of the C-C bond are smaller in the direction of the bond (\parallel) than that normal to the bond (\perp) ($\chi_{\parallel} < \chi_{\perp} < 0$), that is, the anisotropic diamagnetic susceptibility defined by $\chi_a = \chi_{\parallel} - \chi_{\perp}$ is negative [11]. The C-C bond therefore tends to align in the direction perpendicular to the applied field. An aromatic ring possesses a large diamagnetic anisotropy due to the ring current induced on the ring. As a result, aromatic rings tend to align with its ring plane parallel to the applied field. If a particle has a size large enough to overcome the thermal disturbance, it aligns under a magnetic field. A number of examples have been reported: organic crystals, protein crystals, carbon fibres, carbon nanotube, cellulose fibres, poly (ethylene) crystallites, etc., undergo magnetic alignment. Inorganic crystals also align under magnetic fields. Magnetic alignment of these materials is a natural result of their anisotropy in diamagnetism and their size that is large enough to exceed thermal energy. In general, crystals possess diamagnetic anisotropy [10]. Unlike the above examples where the parameters such as the anisotropic susceptibility and the size and the shape of the aligning particle are fixed, there is another class where these parameters are not fixed but change in time, which complicates the alignment behaviour. A typical example is found in the magnetic alignment of crystalline polymers during the crystallization process, where the anisotropic structures responsible for the magnetic alignment may change in size and shape in time. Discussion has been made on structure formation during polymer crystallization [12 - 14].

Mechanical properties are of relevance for all applications of polymers in industry, medicine, household, and others. The improvement of properties in general and the better fitting of specific properties to defined applications is a continuous target of polymer research. Of particular interest is not only the enhancement of the special properties themselves, such as stiffness, strength or toughness, but also the combined improvement of usually contradictory mechanical properties (like strength and toughness) in combination with other physical properties. The defined improvement of the mechanical properties demands a better understanding of the multiple dependence between molecular structure, morphology, polymerization and processing methods on the one hand, and ultimate mechanical properties, on the other; i.e., structure-property correlations. The application of a magnetic field at certain stage of polymer matrix reconstruction may introduce better properties to the final physical state of the polymeric material.

The current work represents a study for the effect of magnetic field on the alignment of polymer chain during its structural change from low inter-attraction forces (liquid phase) to the higher chain inter-attraction forces (solid phase). During this phase transition process other processes may occur too, like crystallization or just chain folding for construction and growth of spherulites. The study will consider polycrystalline polymers as well as glassy polymer. Also, this study will consider magnetic fields from strong permanent magnets instead of the usually used electromagnets.

Provide examples of relevant academic disciplines for this journal: E.g., History; Education; Sociology; Psychology; Cultural Studies;

Experimental techniques:

1 – The applied magnetic field.

The used magnetic field in this work is generated from a set of powerful neodymium permanent magnets manufactured by Applied Magnets Co., USA. Two different magnet shapes were used, disc NdFeB magnets with 4 in. diameter, 1 in. thickness and pull force of 590 lbs. (type N42), and block magnets 2 x 2 x 1 inches with countersunk hole and of 250 lbs. pull force (type N50). Figure 1, shows samples of the used magnets.



Figure 1: Samples from the used permanent magnets

2 – Spherulite growth in magnetic field.

When a polymer crystallizes from the melt without disturbance, it normally forms spherical structures that are called “spherulites” [15,16]. The dimensions of spherulite range from micrometers to millimetres, depending on the structure of the polymer chain and the crystallization conditions, such as cooling rate, crystallization temperature, and the content of the nucleating agent. In the current work the polymer melt is disturbed by a magnetic field in order to show its effect on the spherulite formation and growth.

A special optical system is constructed to record the formation and growth of polyethylene glycol (PEG) spherulite as seen between two crossed Polaroids. Figure 2, is a schematic diagram for the used system, where:

is a light source

- 1 collimating lens
- 2 linear polarizer
- 3 Stand
- 4 Neodymium permanent magnet
- 5 Glass slid
- 6 Glass slid
- 7 PEG sample
- 8 Objective lens
- 9 Telescopic tube
- 10 Linear polarizer
- 11 Cylindrical adapter
- 12 Video camera

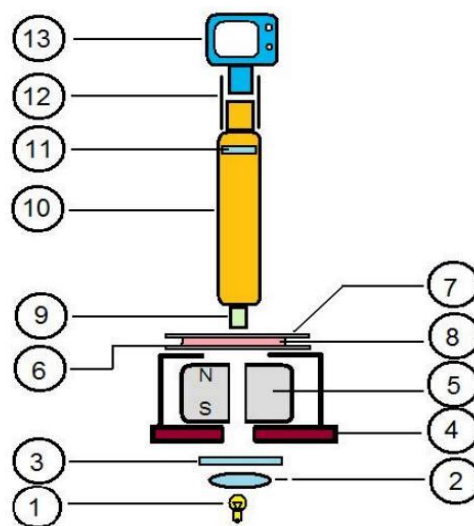


Figure (2): Optical system

The polarisers (3) and (11) are adjusted to be crossed for always, and then no light reaches the camera after focusing it. The sample is placed between two glass slides and heated until the melting temperature. The melted sample is placed on the stand (4), and then the camera is turned on. Two video clips are recorded for the same sample, one without the magnet and the other with the magnet placed just under the sample. The strength of the magnetic field just under the objective lens and above the sample was measured by a digital Tesla meter and was found 355 mT. In this case the lines of magnetic force are crossing the sample (perpendicular to sample surface). The magnet can be placed beside the sample on the stand so that the lines of force are now tangential to the sample’s surface, and a third video clip was recorded where the field strength in this case at the objective lens was 100 mT. Any of these video clips was expanded into its frames. The diameter of a selected spherulite is measured several times and the average is considered. These measurements are carried out for each frame, and then the average diameter is plotted against the time of the frame and hence the rate of growth could be determined. Figure (3), is a typical copy for a frame from PEG spherulite growth clip.

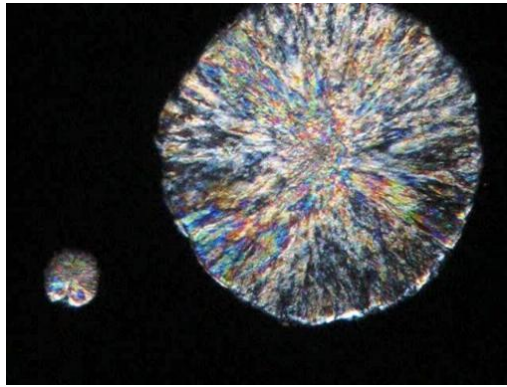


Figure (3): Image for PEG spherulite as recorded in a frame

3 – IR- spectrum of the samples:

In order to examine any chemical changes in a polymer due to external treatment, the infra-red spectroscopy is ideal technique. In the present work, an instrument model NICOLET- IS10 from Thermo Scientific Co., USA is used. Samples from each polymer were examined before and after exposure to magnetic fields with different strength for comparison and deciding the molecular changes, if any.

4 – X- ray diffraction technique:

X - ray diffraction is a powerful tool for the identification of the crystallization state of solids. The crystallization state includes degree of crystallization, crystal size, crystals type, rate of crystallization and crystal distribution as special case. The X-ray diffraction charts may provide well information about the macro-molecular orientations. The mean orientation factors (k) were calculated as the ratio between intensity of crystalline maxima for samples treated in magnetic field and untreated analogues [17]. The x-ray tube that used in the present work was adjusted at the following measurement conditions: Cu target, voltage of 40 kV at 30 mA. (Shimadzu6000, Japan).

5 – DSC and DTA – Thermal analysis:

Differential Scanning Calorimetry (DSC) technique is widely used to investigate the properties of polymers upon heating on certain range at specific rate. Melting and crystallization are examples of phase transitions of the first order. This means a transition where the heat capacity as a function of time and temperature exhibits a discontinuity, which has the shape of a peak. The area under the peak in the thermo-gram is directly proportional to the energy that is emitted or absorbed during changes of state in the sample. The highest point in the peak reflects the temperature or time when the largest part of a sample is melting or crystallizing or when a polymerization or curing reaction occurs at its highest rate. Differential thermal analysis (DTA) is also used when the melting point is the task. In the present work, the Shimadzu50 (Japan), instrument was used in atmosphere of nitrogen and gas flow of 20 ml/min for sample of weight 10 mg in aluminium pan.

6 – Mechanical Stress – Strain Characterization:

There are three factors determine whether a polymer is glassy, rubbery or fibre-forming under a given set of conditions. These are the chain flexibility, the chain inter-attraction and the regularity of the polymer. The relationship has been expressed diagrammatically by Swallow [18]. Failure of polymers in certain applications to carry design loads or occasional accidental overloads may be due to excessive plastic deformation resulting from the inadequate strength properties of the polymer. For the quantification of such failures, the mechanical property of primary interest is the yield strength and the corresponding strain. The ultimate strength, along with the associated strain, also provides useful information [19]. The machine used to measure the recent mechanical properties is Zwick Tensometer Z010, Germany, with calibrated load cell at temperature of $23 \pm 0.2^\circ\text{C}$ and R.H. of $45 \pm 5\%$.

7 – Materials:

In this study four different polymers are selected each for specific feature. All of these polymers are heated until melting and then exposed to a magnetic field with measured strength and left under field until solidification. One of these polymers is solidified from molten phase and from dilute solution. The selected polymers are:-

- a) Polyethylene Glycol (PEG), which is melted at lower temperature and forms a well characterized spherulites.
- b) High density Polyethylene (HDPE), which has long continuous chain, melted at relative low temperature and polycrystalline.
- c) Polypropylene (PP), which has one small side branch and highly crystallizes.
- d) Polystyrene (PS), which is a glassy polymer and it can be processed to form films from its molten and also from its dilute solution in suitable organic solvent.

8 – Films treatment by magnetic fields:

Films from PEG, HDPE, PP and PS are prepared by heating each polymer between two non-ferromagnetic metal plates until the melting point of each, and then the plates are compressed to certain thickness. The plates are then transformed soon and placed on the top of the permanent magnet set after measuring the field strength at the middle point of the holding stage. The film is left above the single pole of the magnet set until cooling to the room temperature (23oC), and then removed from between the metal plates. Another set of films are prepared away from magnets as reference samples. A glass dish containing solution of PS dissolved in methylene chloride is placed above the magnetic pole and left until complete vaporization of the solvent and the film formation. Several films from each polymer are prepared at different field strength by changing the distance between the film and the most top of the magnet set.

Results and Discussion:

Many researchers have decided that application of external magnetic field to a polymer can initiate structural changes which cause improvements in many properties of the polymer. Most of these studies correlated the size of change to the strength of the applied magnetic field disregarding the initial structural state at the moment of field application. In the present study, the author is suggesting that the magnetic field effects are efficient if the field is applied at the lowest chain inter-attraction force. The following discussion is a trial to establish this suggestion; therefore the liquid phase was selected to start the field application. Due to the high temperature of the polymer molten, the molecular system entropy is high which disturbs the chain alignments by the applied magnetic field. Because of this thermal disturbance, a dissolved polymer was tested too. On the other hand, not too strong magnetic fields were used as produced at one pole of a set of permanent magnets. Of course stronger magnetic fields may induce greater structural changes.

The first detected change was the PEG spherulites rate of growth. Video clip for the growth of PEG spherulites is recorded for samples without field and under field application. The field was projected on the sample perpendicular and then tangential to the sample surface. The clip is expanded to its frames and the average diameter of the spherulite was measured on screen and then plotted against time of the frame. Figure 4(a-d), shows four frames at different times for the same sample to clarify the ease of detection of PEG spherulites growth. Figure 5, represents the plot of average spherulite as function of growing time, from which the rate of growth can be determined.

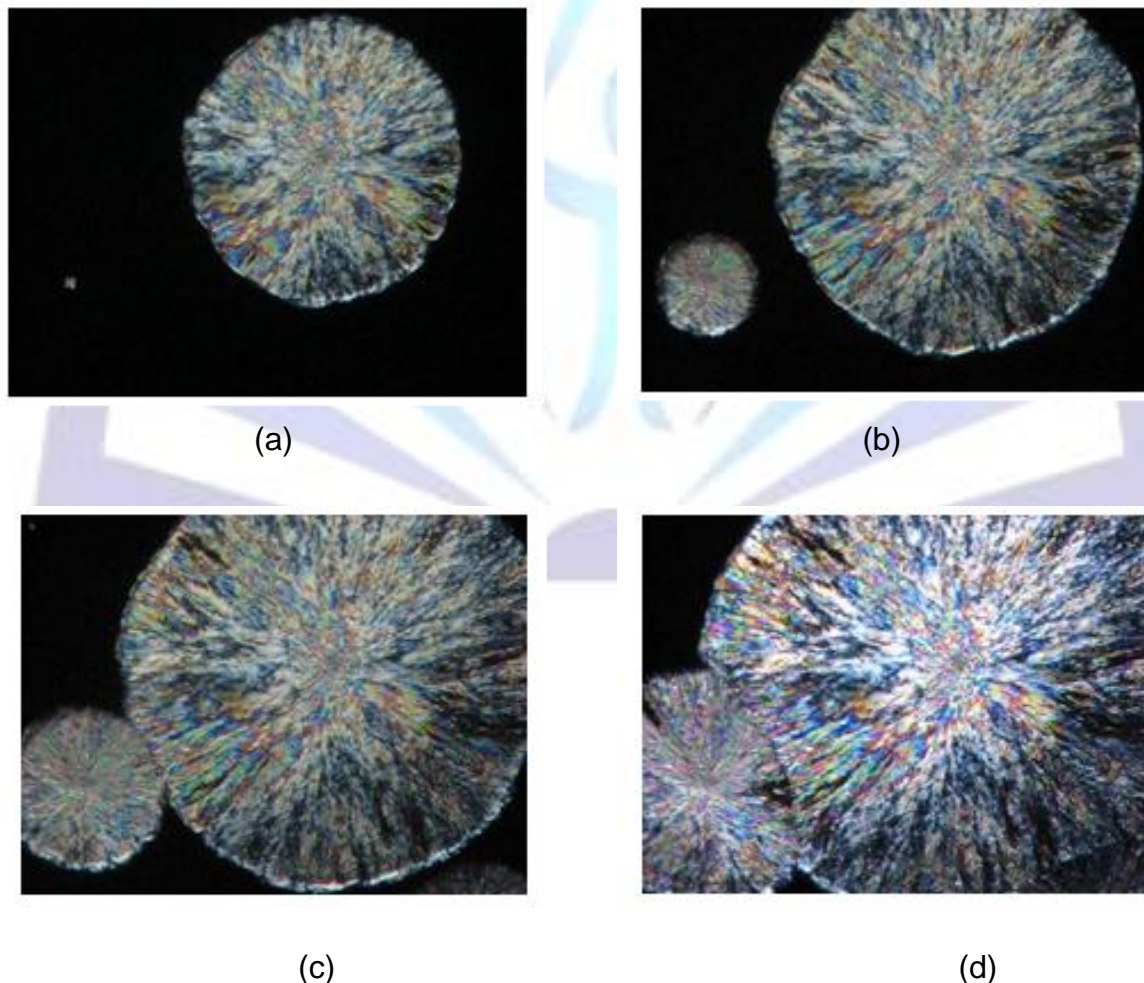


Figure (4 a-d): The growth of PEG spherulites by cooling to room temperature

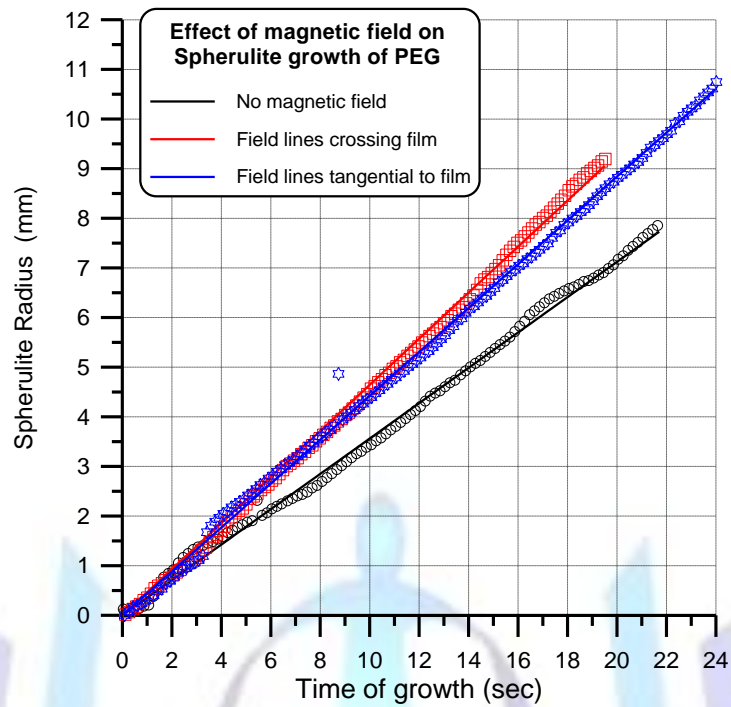


Figure (5): Effect of the magnetic field on the rate of growth of PEG spherulite

From figure 5, one may deduce that the rate of PEG spherulite growth is independent of the direction of magnetic field lines of force with respect to the plane of the film, where the difference between the parallel and perpendicular directions is too small. In fact the effect of tangential field may be greater than the effect by the normal field because the strength of tangential field at the focal point was 100 mT while the strength of the normal field was 355 mT at the same point. Comparing these rates with that of the free growing spherulite leads to that, the magnetic field accelerates the spherulite growth, especially in the direction parallel to the lines of the field.

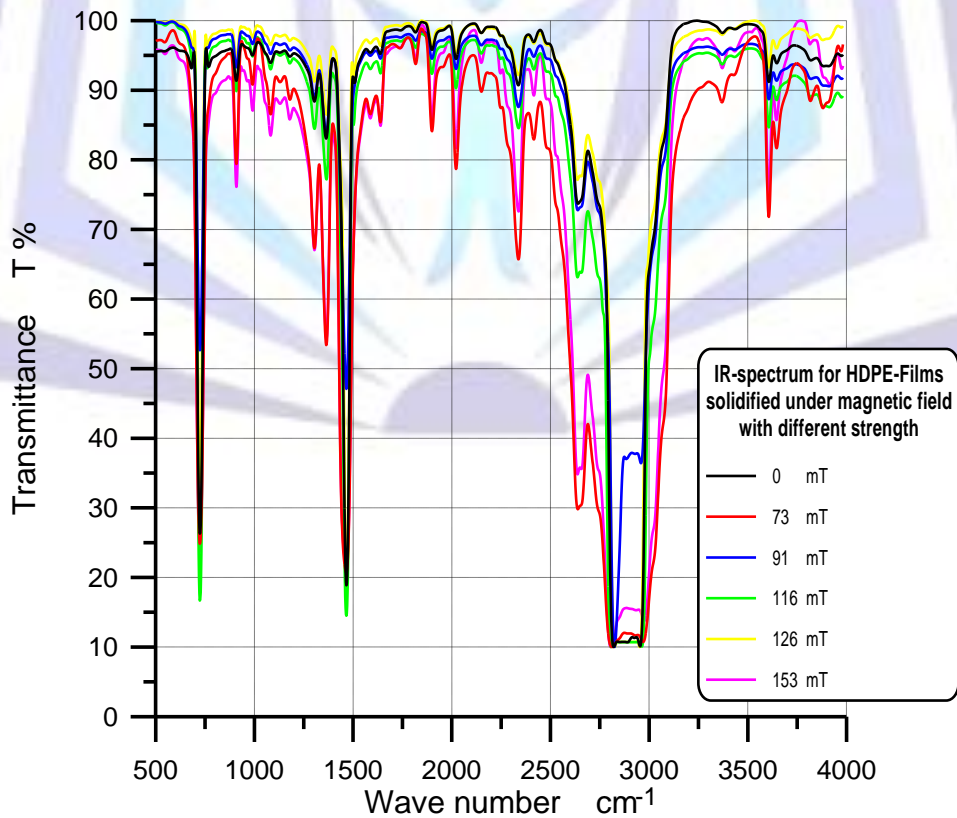


Figure (6): IR-spectrum of HDPE films when affected on by a magnetic field with different strength during transition from liquid to solid phase.



The IR – spectrum of the HDPE films that are solidified in a magnetic field show no serious chemical changes or bonds break. Also there was no appearance for new modes of vibrations. The magnetic field effect may be summarized in broadening of the absorption band at 2500 – 3100 cm^{-1} which may be due to CH₂ symmetric stretch and CH₂ asymmetric stretch [20]. That broadening occurs at field strength of 73 and 153 mT only, which means that the response of the HDPE molecular structure to the applied magnetic field is selective. This means that weak magnetic fields may show equal or higher structural changes than that by stronger fields.

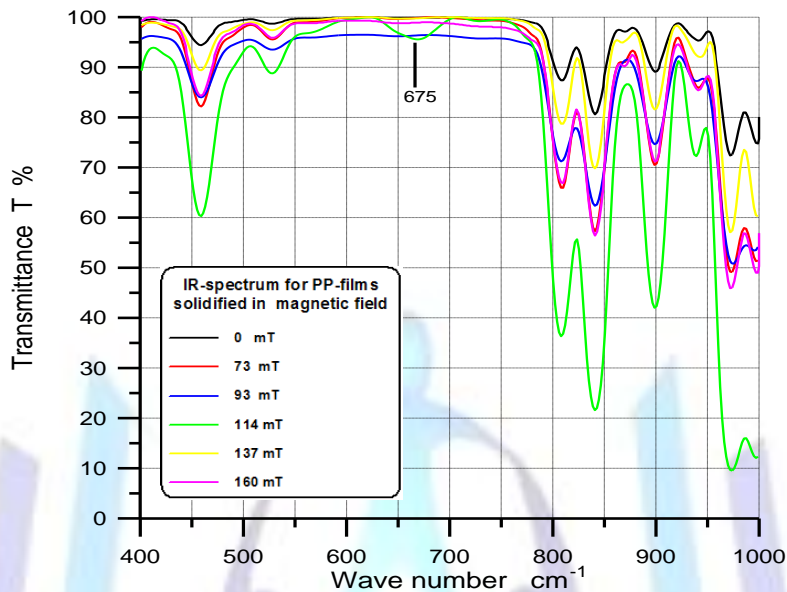


Figure (7): IR – Spectrum for PP – films constructed under effect of magnetic fields with different strength.

Polypropylene films that are solidified under magnetic field show nearly the same responses as HDPE, except appearance of a new weak absorption band at frequency of 675 cm^{-1} only in the spectrum of PP sample that exposed to field of strength 114 mT. The PP sample exposed to 114 mT shows stronger absorption through the whole spectrum. The known IR - spectral lines don't include that new band. This low energy band may be due to the increase of isotacticity of the used isotactic PP by the magnetic field.

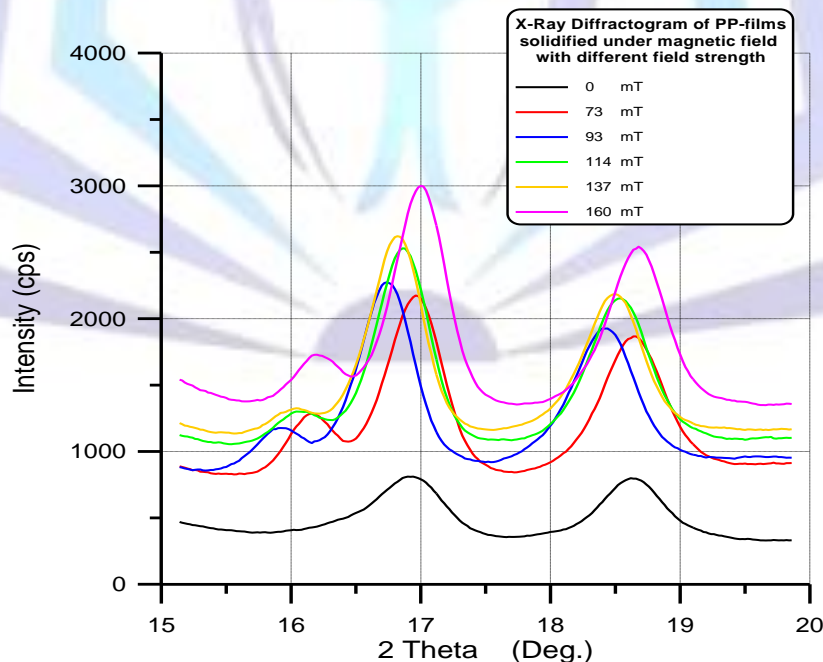


Figure (8): The effect of magnetic field on the crystallization of PP-films during the film transition to the solid phase.

Figure 8 shows formation of new crystallites at the diffraction angle around $2\theta = 16^\circ$ in the PP- films due to the exposure to a magnetic field during solidification (and hence crystallization). It is also clear that crystallites are formed with nearly the same size irrespective to the applied magnetic field strength, which means that weak magnetic fields can initiate

structural changes in PP during its phase transitions. Appearance of such new crystallites may not increase the whole crystallinity of the film. The formation of more crystallites should be started after establishment of high alignment. Figure 9 shows the average orientation factor k as calculated from the X-ray diffraction peaks intensity, as the ratio of a characteristic peak intensity of magnetically treated sample to the intensity of the same peak of the un-treated sample.

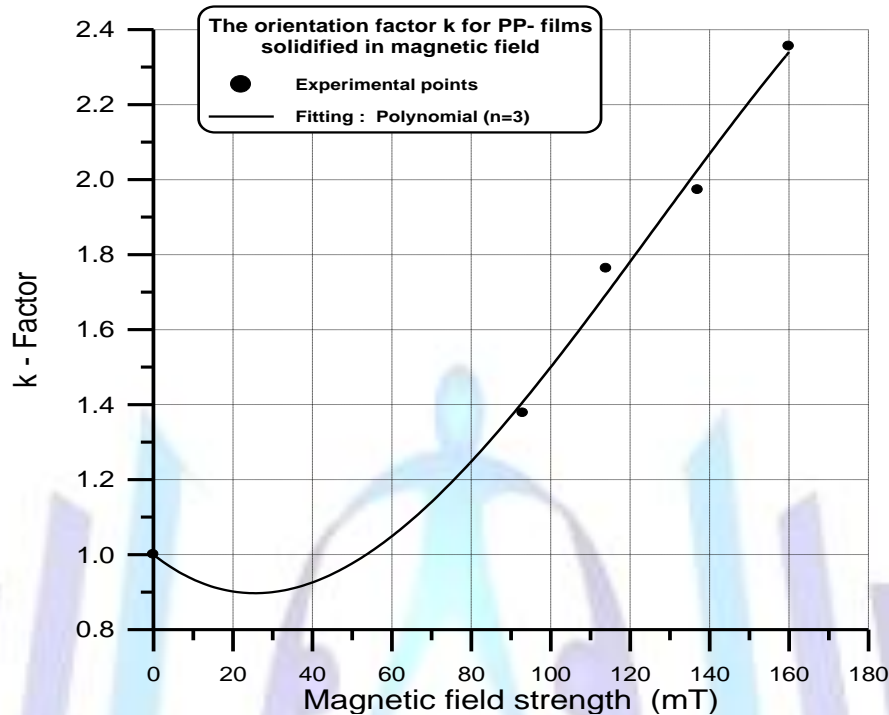


Figure (9): The orientation factor k as function of the applied magnetic field strength on PP- film during its solidification.

In order to examine the effect of film thickness on the calculation of the k – factor, this factor was re-calculated after dividing the peak intensity by the film thickness in microns and the relation in figure 9 is re-plotted between the field strength B against the k -factor per unit thickness, the result is shown in figure10.

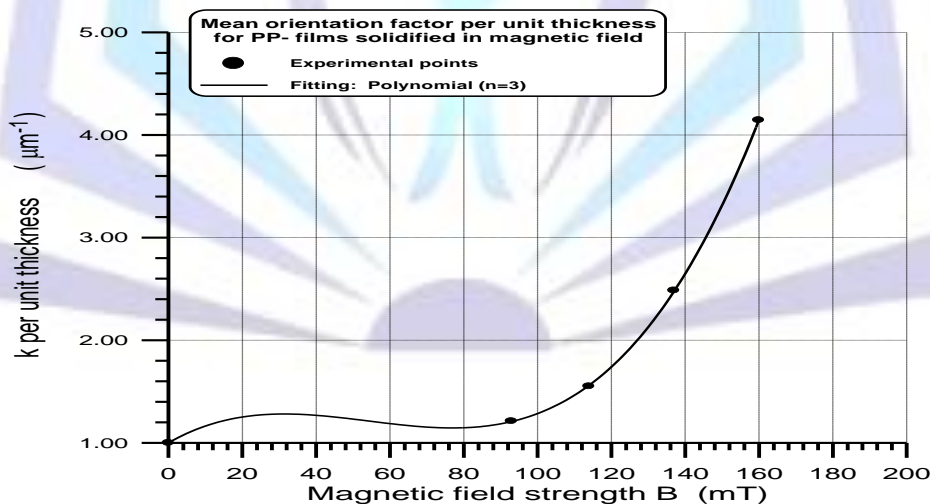


Figure (10): The mean orientation factor- k per unit thickness for PP-films as function of the strength of the applied magnetic field during the film solidification.

It is clear that PP – films possess higher chain alignment as the applied magnetic field increases. Comparing this PP behaviour with the chain alignment by applying a magnetic field on HDPE films, that are prepared with the same technique as shown in figure 11, one may relate the observed behaviour to the presence of the side group $-\text{CH}_3$ in PP structure. This group, when rotated, causes markedly displacement in the centre of mass of the chain due to its high associated inertia, which is not possible in HDPE chains.

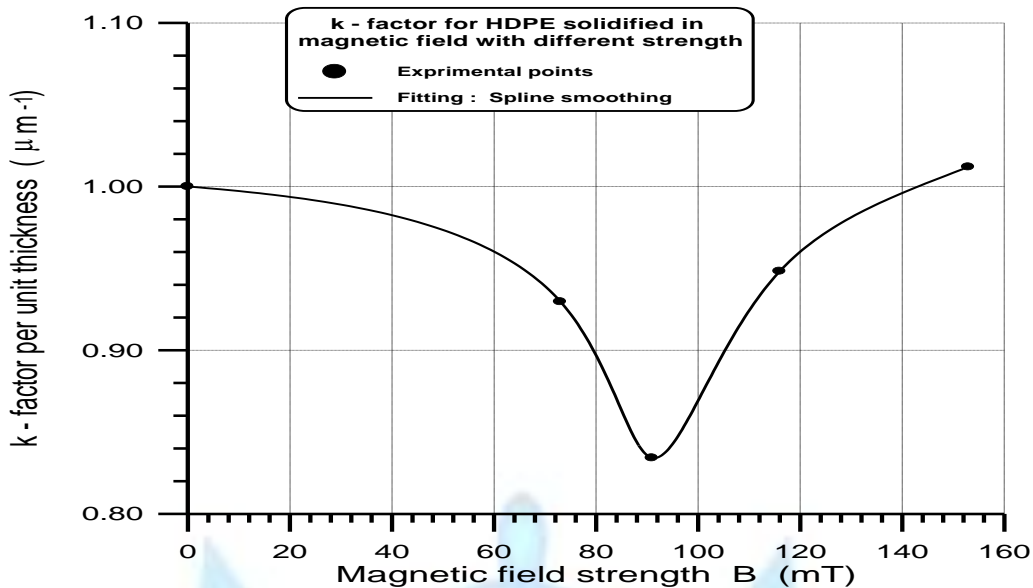


Figure (11): The mean orientation factor-k per unit thickness for HDPE -films as function of the strength of the applied magnetic field during the film solidification.

The behaviour shown in figure 11, clarifies that application of magnetic field on melted HDPE disturbs the low chain orientation to lower state and then the orientation is recovered at higher field strength, which may be due to stretching the zigzag construction of the HDPE chains.

Polystyrene (PS) films that are casted from dilute solutions show opposite structural response to the applied magnetic fields. Figure 12 shows values of the mean orientation factor k of PS films casted from dilute solution in magnetic fields at different strength. The graph shows the enhancement of the mean orientation, and then the polymer structure recovers its original orientation at higher magnetic field strength. PS – films that are prepared by hot pressing the polymer and then left to cool in magnetic field show a same trend as the HDPE films.

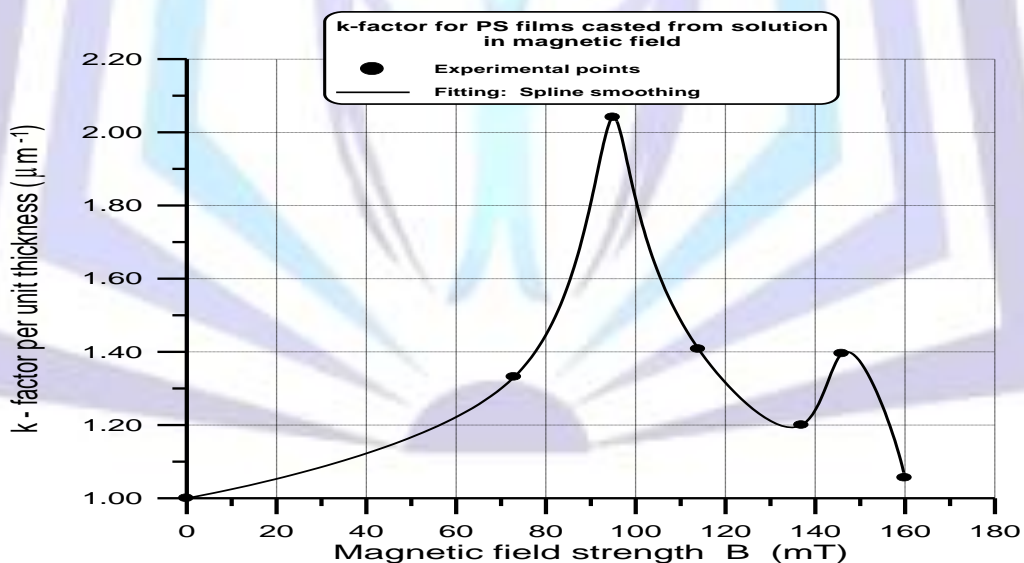


Figure (12): The mean orientation factor-k per unit thickness for casted PS - films as function of the strength of the applied magnetic field during the film solidification.

Two reasons may be behind this contrasted behaviour of PS chain alignment by the application of magnetic fields. The first is the very weak chain inter-attraction force when the polymer chains are extended in the used solvent, and hence the induced magnetic force on chains becomes able to align chains. The second is the longer duration of dissolved PS in the magnetic field until solidification than duration of solidification of melted PS in the same magnetic field. Figure 13 shows that the PS film casted from dilute solution is longer range ordered than the hot pressed film even in absence of fields. This graph supports the effect of longer time of solidification on the orientation of PS chains.

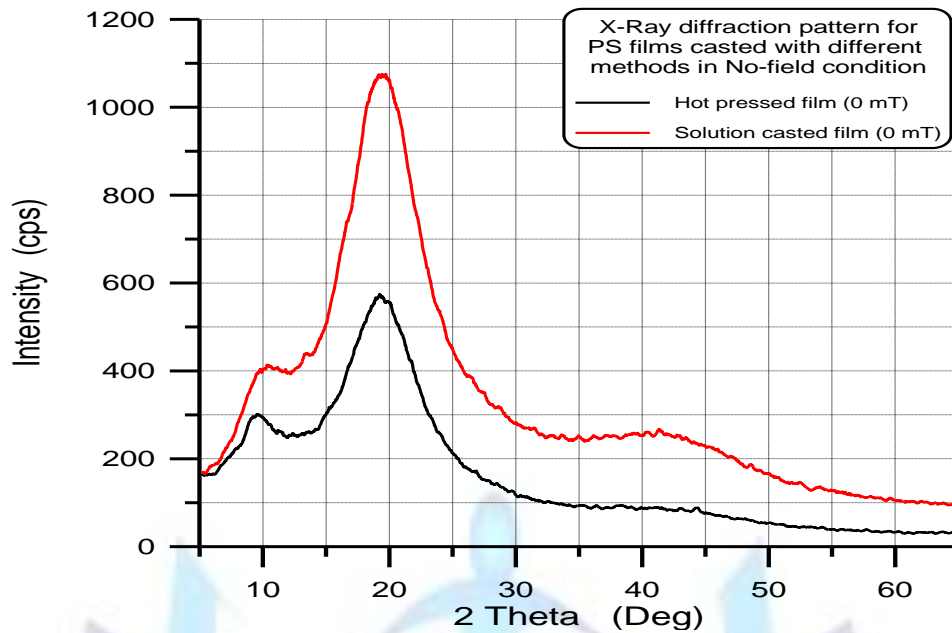


Figure (13): X-ray diffraction of PS – films prepared by two different methods.

It is well known that any variation in the polymer structure should associate changes in some other physical properties. Re-alignment of polymer chains by application of magnetic fields may cause changes in thermal properties of the polymers. Figure 14 shows that application of magnetic field on HDPE during solidification don't affect the melting point of HDPE, only the melting temperature is hesitated between 131 and 136°C.

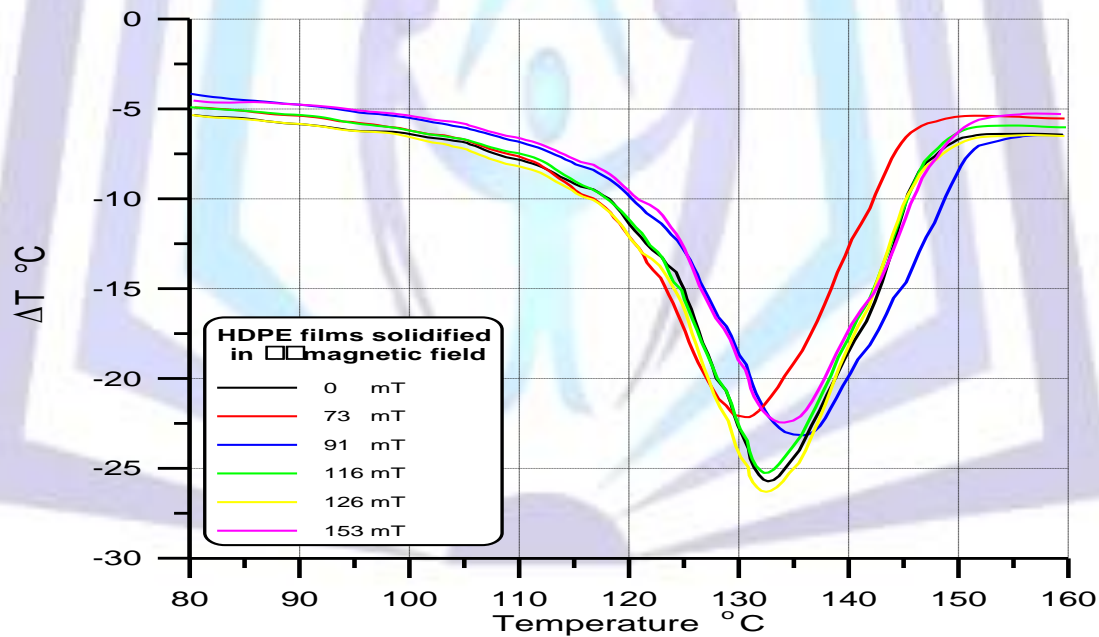


Figure (14): DTA thermo-gram of HDPE films solidified in magnetic fields of different strength.

This behaviour was expected where the IR-spectrum of the magnetically treated HDPE films showed no serious changes in absorption bands. (See figure 6). Application of stronger fields may cause markedly changes. Figure 15 shows the melting point of magnetically treated PP films as measured by the DTA technique. It is clear that the melting point for all magnetically treated samples is shifted about 10°C lower than that of the virgin sample. This shift may be due to the formation of crystallites by the magnetic field. Also, the sample that subjected to the field of 114 mT strength showed additional shift down to about $152 \pm 0.2^\circ\text{C}$ while the virgin sample melts at about $170 \pm 0.2^\circ\text{C}$. This additional reduction may be due to the magnetically induced structural variation in this sample that is represented by the absorption band at 675 nm in figure 7.

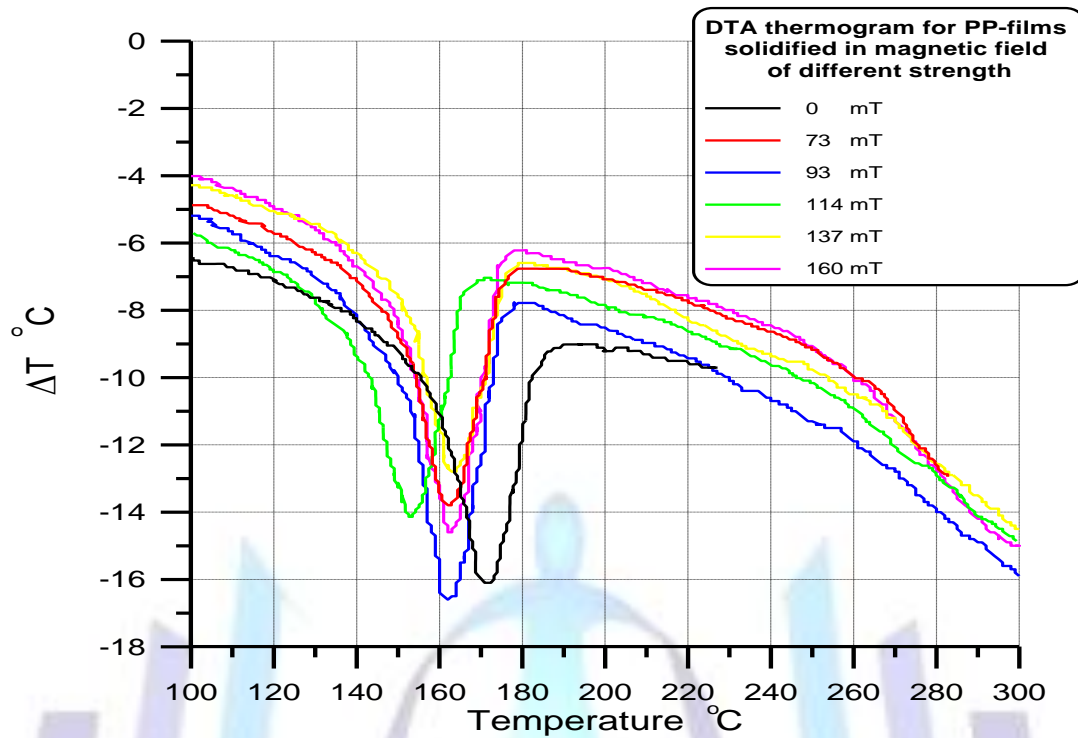


Figure (15): DTA thermo-gram of PP films solidified in magnetic fields of different strength.

Comparing figures 14 and 15 one notes that melting peak of the HDPE is broader than the melting peak of the PP because HDPE is of less crystallinity than PP. Therefore DTA measurements are not valuable for testing amorphous polymers where there is no definite melting temperature. DSC measurement is more valuable in this case. Most of glassy polymers are amorphous; hence the DSC analysis can measure the transition temperatures that are characterizing their thermal properties. Figure 16 and figure 17 represent the DSC thermo-grams of PS films that are casted from dilute solution and from compressed molten and solidified in magnetic field, respectively.

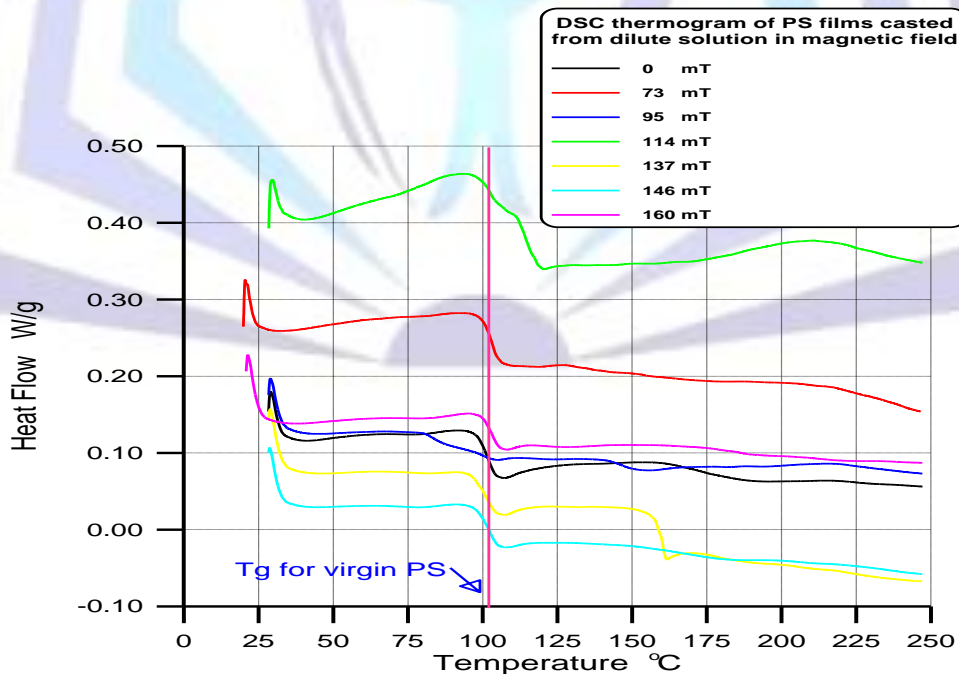


Figure (16): DSC thermo gram Of PS films casted from dilute solution in magnetic fields with different strength.

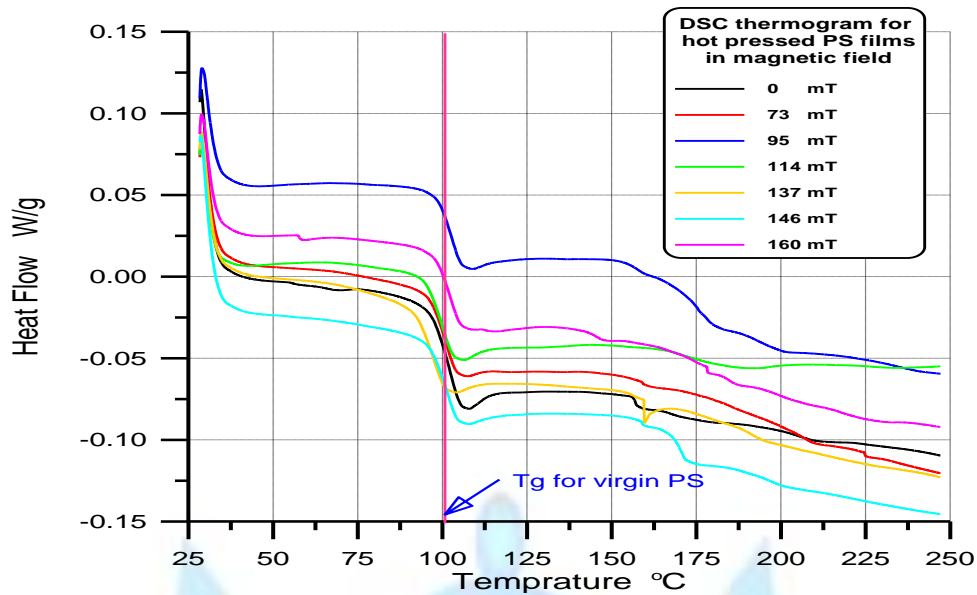


Figure (17): DSC thermo gram Of PS films casted from pressed molten in magnetic fields with different strength.

From these graphs some notes can be drawn:

- a) The glass transition temperature (T_g) of the solution casted PS samples is hesitated around 100°C showing little effect due to the applied magnetic field, while T_g for the pressed molten PS do not show any changes due to the applied magnetic field.
- b) The sample which effected by the field of strength 137 mT showed a remarkable additional transition temperature at about $162 \pm 0.2^{\circ}\text{C}$ in both methods of preparation.

The goal behind many researches in the field of polymer science and engineering is to increase the mechanical strength of polymer products. External effects applied to the polymer are widely followed including the strong magnetic fields. The selection of material phase to be affected on should be additional factor for enlargement the expected enhancement of the mechanical strength. Figure 18 represents the stress – strain behaviour of HDPE films that are solidified in magnetic fields of different strength. The graph shows no markedly variation in the yield stress or strain, which confirms the previous measurements for this polymer in the selected range of magnetic field strength. The stress – strain graph for polypropylene films, figure 19, shows that all magnetically treated samples have mechanical yield strength lower than that of the virgin sample. This trend of the PP mechanical properties confirms the DTA of same samples (see figure 15), where mechanically soft polymers are of lower melting temperatures.

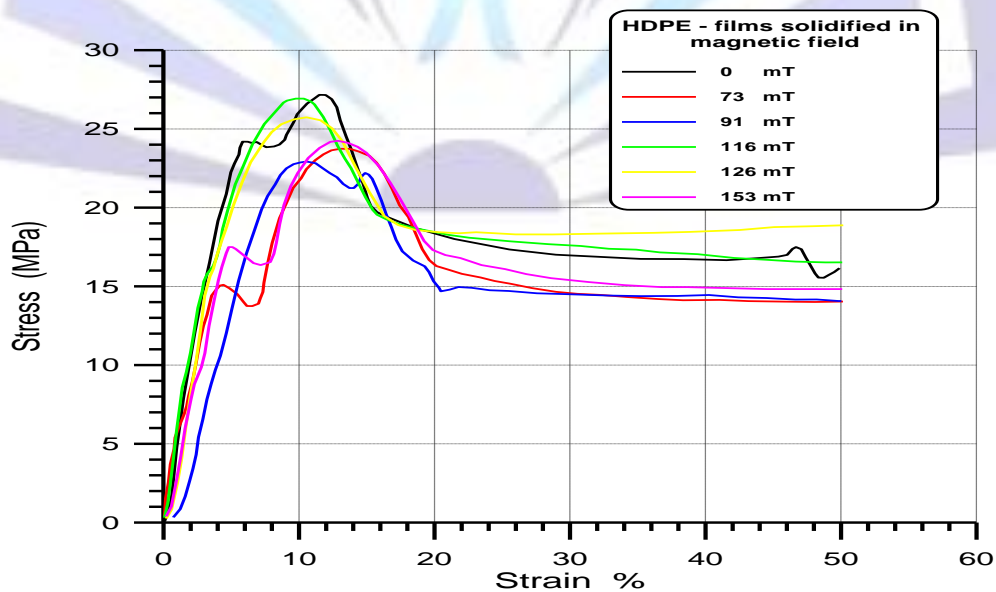


Figure (18): Stress – strain graph for HDPE films in magnetic fields.

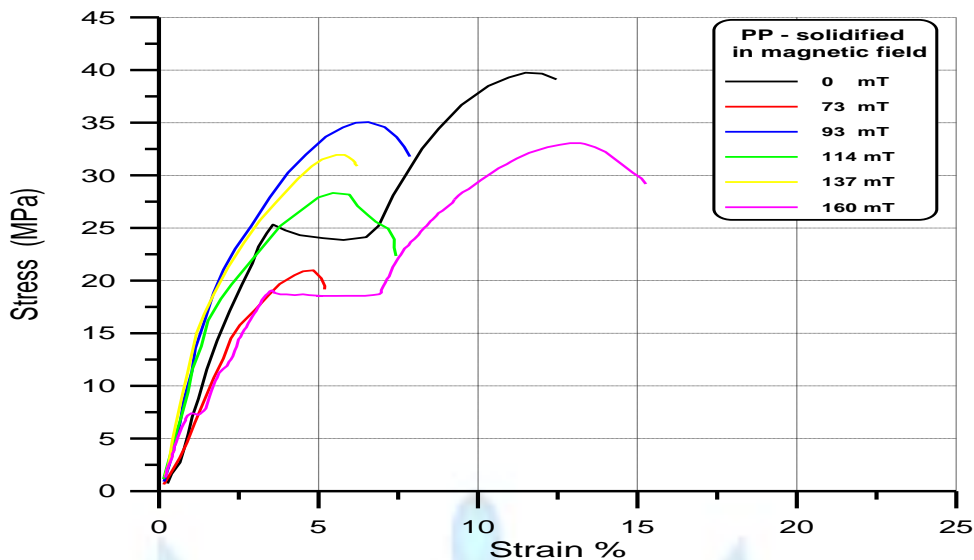


Figure (19): Stress – strain graph for PP films in magnetic fields.

Recalling figure 10, it is clear that the average alignment increases by increasing the magnetic field strength, and hence it should increase the mechanical strength of the polymer which in contrast with obtained data. In fact, it is a fake contrast because the field crosses the sample normally, hence aligns the chain segments normal to both surfaces of the sample, while the tensile measurements are carried out on samples parallel to the surface, i.e.; in direction normal to the oriented chains where the tensile properties are the lowest, as obtained. The results obtained in figure 5 supports this deduction where the effect of a magnetic field on polymer morphology depends on the direction of the field lines as well as the strength of the field.

Conclusion

Application of external fields on a polymer is common physical treatment for modifying the polymer properties. Strong magnetic fields (up to 10 Tesla) were applied either on the final stable polymer or during the polymer synthesising. The present work aims to perform modification in polymer structure at lower magnetic field strength by careful selection of structure state at which the field should be introduced. This study led to the following meanings:

- A magnetic field of strength less than one Tesla can perform remarkably variations in polymer chain structures when the field is applied during the transition from liquid phase to the solid phase.
- Powerful permanent magnets can provide satisfactorily fields with less power consumption and more field stability.
- Many thermoplastic polymers either polycrystalline or glassy show structural variation by application of magnetic fields during their phase transition, but with different responses.
- Polycrystalline polymers with chain side groups (like polypropylene) showed higher response to the applied magnetic fields, while linear or zigzag chains showed less response.
- The time required for complete phase transition is important factor affecting size of the magnetically induced structural variation, as shown in the case of polystyrene casted films with two different methods.
- The direction of magnetic field lines with respect to the film surface is an important factor, too. The rate of spherulites growth in PEG films is a clear example.

Additional studies can be considered for quantitative determination for the dependence of structural variations of polymers on the time of complete phase transition. The effect of magnetic field on the cross-linking of a polymer by electromagnetic radiation is another study worth to be taken in consideration.

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