EFFECT OF COLD DRAWING AND THERMAL AGING ON MECHANICAL AND THERMAL PROPERTIES OF CALCIUM CARBONATE FILLED POLYPROPYLENE BLOCK COPOLYMER COMPOSITE

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Abstract - In this experimental study is done in order to see the effect of Cold Drawing and/or Thermal Aging on the mechanic and thermal characteristics of pure Polypropylene Block copolymer (PPB) and PPB Composite materials with Calcium Carbonate (CC). Every passing day usage of the plastic and composite pipes for transportation of cold or hot, clean or waste, pressured or non-pressured water is increasing. Transportation systems with plastic and composite pipes are expected to be longevous, reliable, economic and ecological. In addition to these factors also rigidity and their resistance in Tensile Test and Charpy Impact Test are demanded to be high. In this concept according to the purpose of this study; uniaxial Tensile and Vicat Softening Point Tests are done to pure PPB and PPB/CC composite materials filled with 10% and 15% CC (by volume) which previously had the Cold Drawing and/or the Thermal Aging operation. The results of the experiments showed that when change on the order of Cold Drawing and/or Thermal Aging operation is done, it influences the obtained features. In general after each combination of operations, increase effect determined on Cold Drawing to Young's Modulus, Thermal Aging to Yield Elongation, Breaking Stress and Vicat Softening Point also while Breaking Strain values are decreasing however Yield Stresses are not affected.

Keywords – Cold Drawing, Thermal Aging, Polypropylene Block Copolymer, Calcium Carbonate.

Introduction

Polypropylene (PP) composite is one of the most extensively produced polymers, especially widely used as automotive parts [1] due to its good impact resistance as well as processability [2]. Polypropylene (PP) is a commodity plastic widely used in food packaging, medical care, automobile and other industrial sectors. Conventional micro fillers such as mica, talc, glass fiber, carbon black, etc. are incorporated to PP in order to further improve its mechanical performances. However, addition of high amount of micro fillers (20–30 wt %) are needed to achieve desired mechanical properties. The additions of large volume content of micro fillers are detrimental to the processability of the composites. [3].

Generally, the dispersion of CaCO3 nanoparticles in PP melt depends on both extrinsic and intrinsic variables. Extrinsic variables include screw configuration, extrusion temperature, screw speed, etc. and Intrinsic variables involve compatibility between PP and CaCO3, viscosity of PP melt, surface free energy of particles, etc. Heat release was clearly felt during cold drawing and typical fibrillation was observed on the fracture surface. Semi-crystalline polymers such as PP were made up of lamellae containing folded chains and they were believed to deform during cold drawing by initial spherulites breakdown and subsequent reorganization of crystalline region [4].

It is well known that the physical properties of polymeric materials are much influenced by molecular orientation. Cold Drawing occurs because at low extension rates the molecular chains in the plastic have time to align themselves under the influence of the applied stress. Thus the material is able to flow at the same rates as it is being strained [5, 6]. Accompanying the molecular processes occurring during the orientation are macroscopic changes in the shape of the tensile specimen [7].

Experimental

Materials

In this study, a Polypropylene Block copolymer (PPB) with commercial name of BorECO™ BEC5015 (Borealis A/S) was used. Density and melting flow rate (MFR) values of this material are 900 kg/m^3 and 0.30 g/10 min (230°C/2.16kg), respectively. Additionally 1500 MPa Tensile modulus, 30 MPa Yield Stress and 10% Yield strain are some mechanical properties of pipe raw material used for non-pressurized pipe systems. These values were taken from manufacturers (Borealis 22.01.2008 Ed.9).

As a filler, Calcium Carbonate (CC) with commercial name of Mikrocarb-5 (Mikro Mineral Endüstriyel Mineraller San. ve Tic. A.Ş.) was used. Its average particle size and density are 5.00 μm and 2700 kg/m^3, respectively. The results of the manufacturer analysis about particle size distribution of this material is given below in Table 1 (Mikro Mineral 2004 Rev:03).

Table 1 Particle Size Distribution

Particle Size	Unit	Typical Value
Average dia. D(0.5)	μm	5.00
TopCut d(0.97)	μm	28.00
Particles under size of 2 μm	%	30
Sieve remaindered of 45 μm	%	0.1

Composite Preparation

Twin-screw extrusion machine with screw length (L/D) value of 38.5, screw length (L) of 2700 mm (Coperion Werner and Pleiderer ZSK 70 Mcc) was used in production line of PPB/CC composite raw materials. PPB/CC composite raw materials filled with percentage of 10 and 15 CC in volume (25.00 and 34.62 % in weight) were produced by physical modification. Production of composite raw materials was performed with vacuum pressure of -1 bar, rotation speed of 750 rpm and production capacity of 500 kg/h, zone temperature range between 220°C and 245°C and cooling water temperature of 30°C. Dehumidification at 50°C for 4h was performed for every composite raw material.

Multi-Purpose Specimens Preparation

Pure PPB and PPB/CC composite raw materials were molded by injection, according to ISO 3167 Type-1A multi-purpose test specimens which have dimensions of a 216 mm x 10 mm x 4 mm injection machine (Arburg Allrounder 370 CMD) with 25 mm screw diameter (D) and 20 screw length (L/D). In molding process which was achieved by considering ISO 294-1 standard, injection parameters; fixed 225°C as melt temperature, 225°C/220°C/220°C/215°C as zone temperature, 1450 bar as injection pressure, 650 bar as holding pressure, 60 s as waiting time and 30°C as die cooling temperature were used.

Multi-purpose test specimens were stored at comfort conditions with no-daylight for at least 24 months.

Tensile Test

Tensile tests were done considering ISO 527-1 with a tensile testing machine (INSTRON 4411 H 4240) which has a capacity of 5 kN with Clip-on static extensometer (INSTRON 2630-111) and Bluehill-2 version.2.6 software on it. According to ISO 3167 (or ISO 527-2) standard 216 mm x 10 mm x 4 mm sized Type 1A tensile samples were placed in the tensile test machine. In test process 1 mm/min. tensile draw speed used to define the Young's Modulus and 50 mm/min. for determining other properties. Tests were done under 20°C ± 1°C temperature and 50±10% humidity and for each value at least 5 test specimens were used. Obtained values were statistically considered according to Chauvenet Criteria [8].

Vicat Softening Point Test

Experiment was done with Zwick Vicat Softening Point Test machine with the weight of 50 N and 50°C/h heating speed.

In the experiments, multi-purpose experiment samples (MPES) with10 mm x 10 mm x 4 mm size in group of minimum 3 are used and these samples are cut according to Method B50 of ISO 360.

Cold Draw and Thermal Aging Implementation Steps

To the MPES, Cold Draw (A) and Thermal Aging (B) operations are applied with different orders or separately. Please see Table 2.

Table 2 Operations and their order applied on MPES

	Materials filled by volume %		
	0	10	15
Step 1	A (only)		
Step 2	B (only)		
Step 3	A (before) + B (after)		

Firstly through Cold Draw operation (Step 1) with 1mm/min speed, %2 strain (ε) on MPES is provided. Afterwards, only Thermal Aging (Step 2) is done by making the pieces wait 50 hours in a 80°C temperature oven. During the experiment contact of MPES with thermal oxidation is blocked by placing them into experiment boxes.

Later on, on the MPES which had %2 strain (ε) through 1mm/min speed Cold Draw operation, Thermal Aging is applied by making them wait 50 hours in an 80°C temperature oven (Step 3).

Results are shown in graphics based on values obtained from pure PPB and PPB/CC composite materials.

Results and Discussion

It should be stated especially that pure PPB raw material was used without any effect from twin screw extrusion parameters, like PPB/CC composite raw materials. Also, both pure PPB and PPB/CC composite raw materials were formed to multi-purpose test specimens by injection molding with constant injection parameters (zone temperatures, heat load amount, etc.).

Uniaxial Draw Test results of pure PPB and PPB/CC composite raw materials which Cold Draw and Thermal Aging operations applied or not applied are shown in Figure 1 to 5 and results of Vicat Softening Point Test are given in Figure 6.

When Figure 1 is examined, it is seen that, thermal aging causes decrease on Young's Modulus however cold drawing increases Young's Modulus. But also when thermal aging is applied after cold drawing, a small reduction on Young's Modulus is determined.

In Figure 2 it is seen that Yield Stresses of the materials are not changed neither when Cold Draw and Thermal Aging operations are implemented separately nor when both operations are applied after each other.

With Figure 3 it is possible to see that Yield strain is affected separately from each operation. Yield strain values of composite materials, with 15% CC in volume, have the highest increase with thermal aging and the biggest decrease with cold drawing.

In Figure 4, it is determined that Breaking Stress values are raised with Thermal Aging operation.

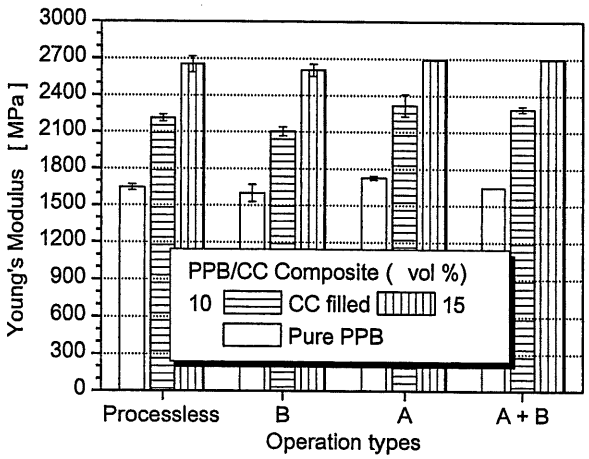

Figure 1 The cold drawing and/or the thermal aging process effect of Young's Modulus

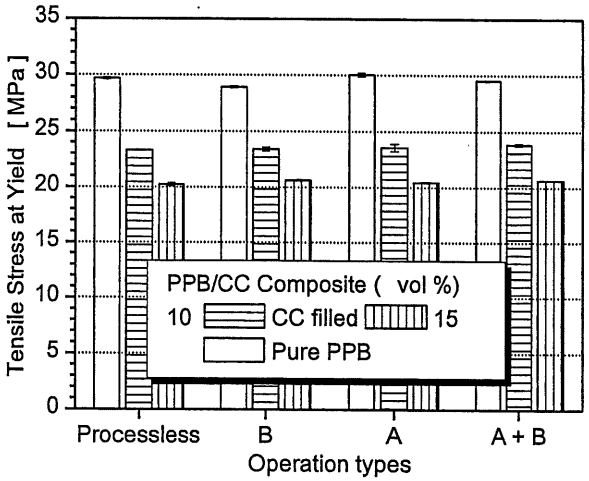

Figure 2 The cold drawing and/or the thermal aging process effect of Tensile Stress (at yield)

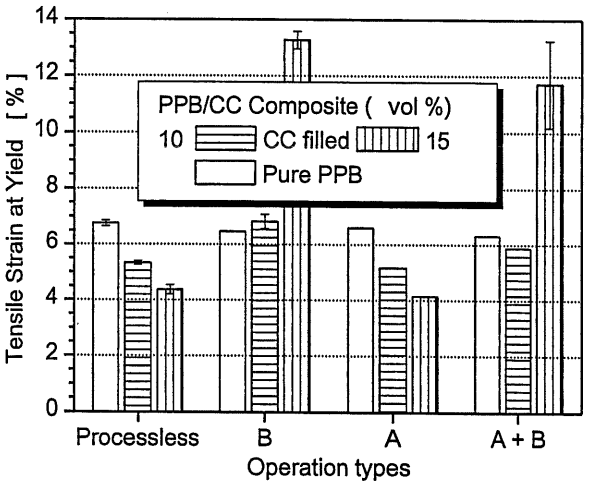

Figure 3 The cold drawing and/or the thermal aging process effect of Tensile Strain (at yield)

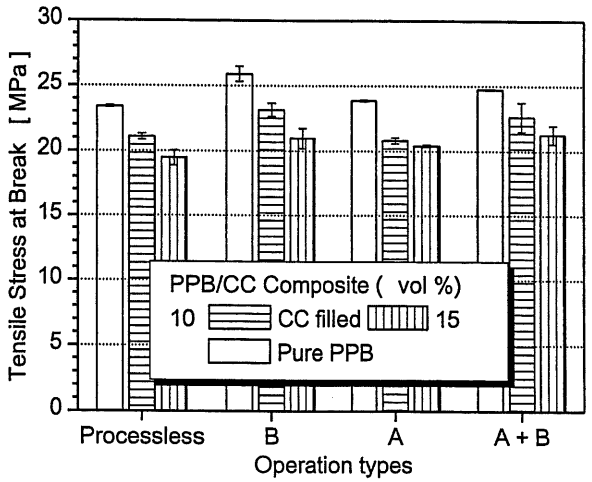

Figure 4 The cold drawing and/or the thermal aging process effect of Tensile Strain (at break)

Breaking Strain of the materials is affected separately from each operation (Figure 5), as it was Yield Elongation of the pieces (Figure 3). It is also determined that Thermal Aging increased the Breaking Strain of only pure PPB materials.

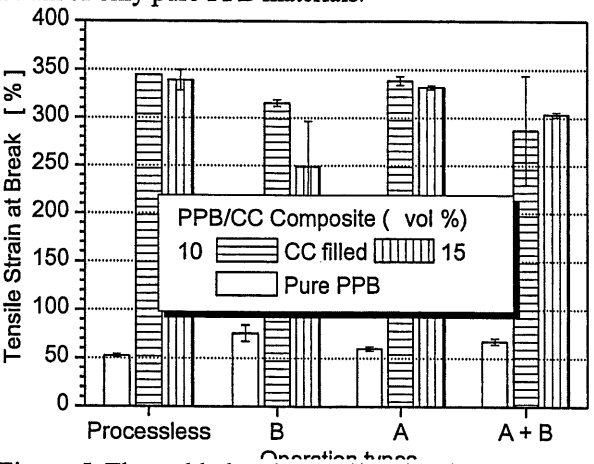

Figure 5 The cold drawing and/or the thermal aging process effect of Tensile Strain at Break

Figure 6 demonstrates that Vicat Softening Point rises with Thermal Aging and lowers with Cold Drawing.

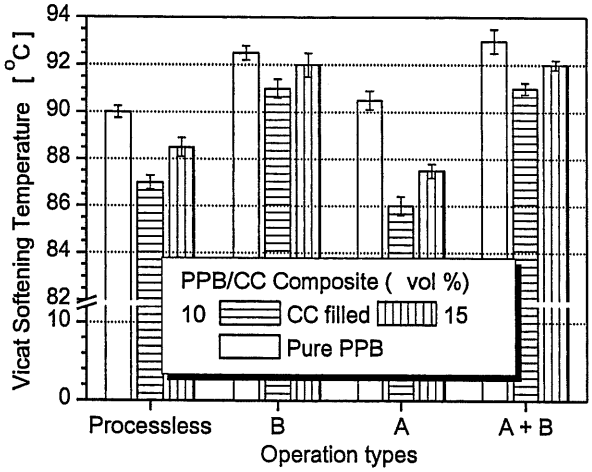

Figure 6 The cold drawing and/or the thermal aging process effect of Vicat softening temperature

Conclusions

It is stated that Cold Draw and Thermal Aging operations have impact on the mechanical properties of pure PPB and PPB/CC composite materials filled with 10% and 15% CC (by volume).

Cold Draw and Thermal Aging caused positive effects on some properties and negative impacts to some other characteristics.

During PPB/CC composite materials studies, selection of smaller averaged grain size and /or CC materials having hydrophobic feature, under different Cold Draw and Thermal Aging parameters should be considered.

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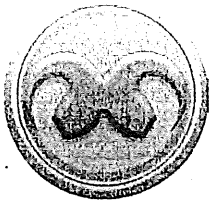

The Polymer Processing Society

The Polymer Processing Society (PPS) was founded in March 1985 at the University of Akron, Ohio, USA. The intent was to provide a mechanism and format for interaction and presentation of research results in the international polymer processing community. The goals of PPS are to foster scientific understanding and technical innovation in polymer processing by providing a discussion forum for the worldwide community of engineers and scientists in the field. The thematic range encompasses all formulations, conversion and shaping operations applied to polymeric systems in the transformation from their monomeric forms to commercial products.

Membership for the PPS is open to all researchers in the field, and to all persons who feel the activities of the society advance their professional development.

PPS website : www.tpps.org

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2nd	Montreal, Canada, 1986
3rd	Stuttgart, Germany, 1987
4th	Orlando, FL, USA, 1988
5th	Kyoto, Japan, 1989
6th	Nice, France, 1990
7th	Hamilton, Canada, 1991
8th	New Delhi, India, 1992
9th	Manchester, UK, 1993
10th	Akron, OH, USA, 1994
11th	Seoul, Korea, 1995
12th	Sorrento, Italy, May 27-31, 1996
13th	New York City, NY, USA, 1997
14th	Yokohama, Japan, 1998
15th	's-Hertogenbosch, The Netherlands, 1999
16th	Shanghai, China, 2000
17th	Montreal, Canada, 2001
18th	Guimaraes, Portugal, 2002
19th	Melbourne, Australia, 2003
20th	Akron, OH, USA, 2004
21st	Leipzig, Germany, 2005
22nd	Yamagata, Japan, 2006
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Inspire AG, Switzerland

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Special Symposia
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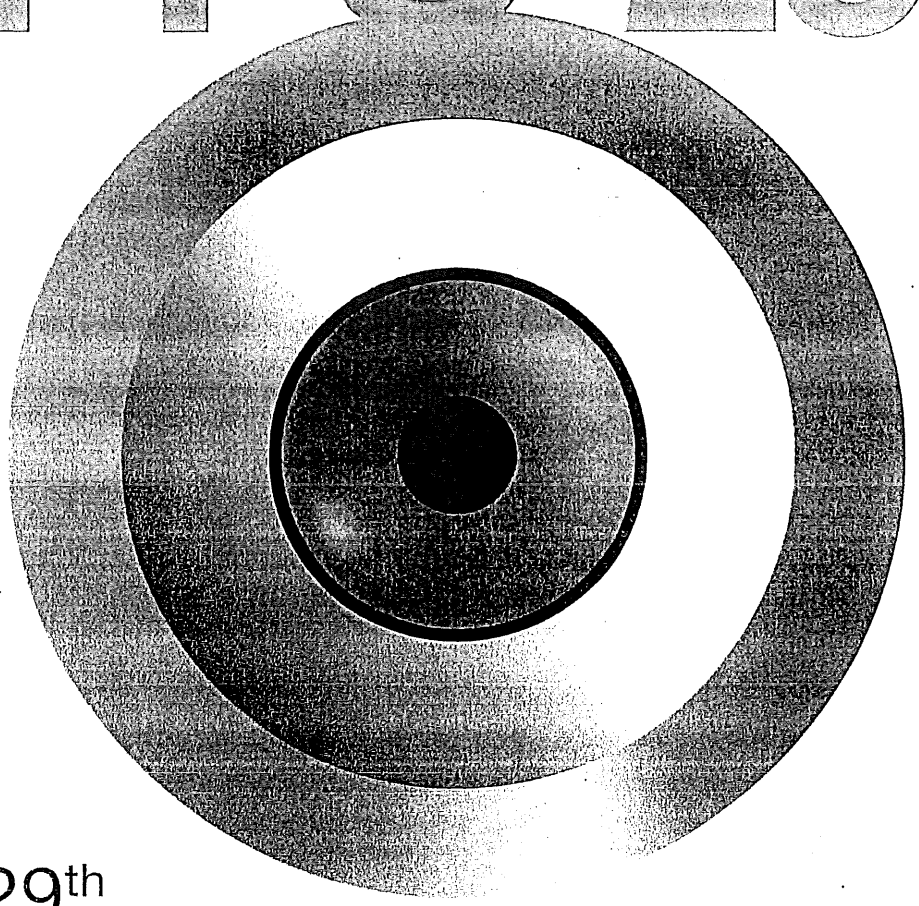

29th International Conference of the

POLYMER PROCESSING SOCIETY

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