Wood and Mineral Fillers for Injection Molding Grade Polypropylene

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ABSTRACT

This paper discusses the results of a filler study conducted by the USDA Forest Service, Forest Products Laboratory, that compared various wood fillers to commercially available mineral and glass fillers in injection molding grade polypropylene. Little documentation exists that directly compares wood and mineral fillers using the same polymers, industrial-scale processing equipment, and standard test methods. This study was intended to provide baseline information for such a comparison. Homopolymer and high impact copolymer polypropylenes were selected to represent the wide range of performance available with unfilled polypropylene. Comparisons were made at both equal weight and approximately equal volume filler loadings. This distinction is important because most minerals have a specific gravity about 2.7 to 2.8, and fiberglass has a specific gravity of 2.5. When compressed to its maximum density by the melt pressures incurred during compounding and injection molding, wood has a specific gravity of approximately 1.4. Therefore, wood displaces about twice the volume of polymer as does minerals at the same weight loadings. The same processing and test equipment were used throughout the study. From our study, we conclude that for the homopolymer polypropylene, wood fillers had similar performance to talc, with lower specific gravity. For the copolymer polypropylene, wood fillers also had similar performance to talc with lower specific gravity, with the exception of unnotched Izod impact performance.

INTRODUCTION

This paper discusses the results of a filler study conducted at the USDA Forest Sevice, Forest Products Laboratory, that compared various wood fillers with commercially available mineral and glass fillers in injection molding grade polypropylene. Use of wood fillers in extruded plastic products is a fairly common pracrice, and injection molding applications are growing (1).

A thorough search of the literature yielded no citations where a comparison was made between wood fillers and mineral fillers using the same polymers, industrial-scale processing equipment, and standard test methods. Our study was intended to provide baseline information for such a comparison. We did not intend to optimize any particular performance attribute or conduct a study of coupling agents. comparabilizers, or impact modifiers. As such, additives were not included in our study. We selected homopolymer and high impact copolymer polypropylenes to represent the wide range of performance available with unfilled polypropylene.

In our study, comparisons were made at equal weight and approximately equal volume filler loadings. This distinction is important because most minerals have a specific gravity about 2.7 co 2.8, and fiberglass has a specific gravity about 2.5. When compressed to its maximum density by the melt pressures incurred during compounding and injection molding, wood has a specific gravity of approximately 1.3 to 1.4. Therefore, wood displaces about twice the volume of polymer as does minerals at the same weight loadings. The same processing and test equipment were used throughout the study.

EXPERIMENTAL

Materials

The formulations we chose for evaluation are listed in Table 1. Formulations were made in either homopolymer or high impact block copolymer polypropylenes. The homopolymer was Solvay 3907[1], with a 36.5 melt index (MI). The high impact copolymer was Montell Pro-fax SB-642 with an MI of 22. Data on the unfilled performance of the polymers are included for comparison.

The wood fillers were either wood flour or demolition wood fiber. The wood flour selected was nominal 40-mesh pine (PI) supplied by American Wood Fibers, Schofield, Wisconsin. Wood flour is typically derived from clean post-industrial sources, such as residue from window and door manufacture. The demolition wood fiber was supplied by Wood Recycling, Inc., Woburn, Massachusetts. Demolition wood (DW) comes from post-consumer recycled sources, such as demolished buildings, old pallets, and urban tree trimmings. It is currently used for hydromulch and a few select composite applications. For purposes of our study, demolition wood was screened to -40 mesh to approximate the physical size of the wood flour.

Minerals were recommended and supplied by their respective manufacturer for the homopolymer. The calcium carbonate (CC) was Optifil-T, supplied by J. M. Huber, Quincy, Illinois,. The “T” denotes treatment with 0.75% to 1.5% stearic acid, used as a dispersing agent. The fiberglass (FG) was 144A, supplied by Owens Corning, Toledo, Ohio. The talc (TA) was VERTAL 710, supplied by Luzenac America, Englewood, Colorado. Variations of these fillers, often with additives, may provide more optimal performance.

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[1] The use of trade or firm names in this publication is for reader information and does not imply endorsement by the U.S. Department of Agriculture of any product or service.

All formulations reported are based on weight percentage. Wood-filled formulations with 20% loadings are intended to approximate the same volumetric polymer displacement as the 40% mineral- and glass-filled formulations. Average list price for truck load quantities, standard packaging (bags or boxes on pallets), f.o.b. location of manufacture, for mid-June 1996, are included for informational purposes in Table 2.

Table 1—Formulations based on weight percentage

Code	Poly-propylene (%)	Polypropylene type	Filler amount (%)	Filler type
PI-20-H	80	Homopolymer	20	Pine wood flour
DW-20-H	80	Homopolymer	20	Demolition wood fiber, screened
PI-40-H	60	Homopolymer	40	Pine wood flour
DW-40-H	60	Homopolymer	40	Demolition wood fiber, screened
TA-40-H	60	Homopolymer	40	Talc
FG-40-H	60	Homopolymer	40	Fiberglass
CC-40-H	60	Homopolymer	40	Calcium carbonate
PP-00-H	100	Homopolymer		
PI-20-C	80	Copolymer	20	Pine wood flour
DW-20-C	80	Copolymer	20	Demolition wood fiber, screened
PI-40-C	60	Copolymer	40	Pine wood flour
DW-40-C	60	Copolymer	40	Demolition wood fiber, screened
TA-40-C	60	Copolymer	40	Talc
FG-40-C	60	Copolymer	40	Fiberglass
CC-40-C	60	Copolymer	40	Calcium carbonate
PP-00-C	100	Copolymer		

Table 2—Average list price of materials f.o.b point of manufacture, mid-June 1996

Material	$/kg
Pine wood flour (PI)	0.176
Demolition wood (DW)	0.242
Talc (TA)	0.270
Fiberglass (FG)	1.784
Calcium carbonate (CC)	0.299
Homopolymer polypropylene	1.06
Copolymer polypropylene	1.64

Processing

Compounding was conducted using a 32-mm Davis Standard Corporation (Pawcatuck, Connecticut) co-rotating, intermeshing twin-screw extruder. The extruder has segmented screws with a length to diameter ratio of 32 to 1. There are eight electrically heated, water-cooled barrel sections, with vent zones at the fourth and seventh sections. Power is supplied by a 15 hp DC drive, and a four-hole strand die is fitted to the discharge end. All materials were compounded with the same screw configuration, but power and feed rates varied somewhat in order to keep melt temperatures relatively equal at about 190°C. Discharge rates were between 20 and 40 kg/hour. Extrudate was cooled in a water trough and cut into pellets.

All materials were dried in an oven at 105°C for at least 24 hours before injection molding into standard ASTM test specimens at 190°C. The injection molder was a 33-ton Cincinnati Milacron (Batavia, Ohio) reciprocating screw type. All materials were molded under the same conditions.

Testing

Testing was conducted according to ASTM standards for plastics (2). Notched and unnotched Izod impact testing were carried out according to ASTM D256. Flexural and tensile properties were determined using ASTM D790 and D638. Shrinkage rates were determined using ASTM D955. Heat deflection temperatures were taken according to ASTM D648 (264 lb/in^2). Melt flow index measurements were performed according to D 1238 with one notable exception. Melt flow index for polypropylene (PP) is usually measured at 230°C, which is well above the thermal degradation point for wood. Therefore, melt flow index for all materials was taken at 190°C.

Scanning Electron Micrographs

Izod impact fractured surfaces were coated with gold and examined using a JSM-840 scanning electron microscope (SEM). The condition of the fibers after processing was also examined by SEM after extracting the polypropylene from the composite specimens in xylene.

RESULTS

Figures 1-6 are scanning electron micrographs of the fracture surface of the Izod impact specimens. They are included to show tiller dispersion, distribution. and fiber length retention. Results of the testing for the homopolymer and copolymer formulations are given in Tables 3 and 4, respectively.

Table 3—Summary of properties and performance: Homopolymer

				Izod impact (J/m)		Tensile properties			Flexural properties		
Formulation	Specific gravity	Melt index[a] (g/10 min)	Mold shrinkage (%)	Notched	Unnotched	Strength (MPa)	Elongation (%)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)	Heat deflection (°C)
PI-20-H	1.00	8.6	0.94	13	110	26.4	4.9	2.3	43.7	1.7	69
DW-20-H	0.99	7.8	0.92	12	130	26.7	4.4	2.3	44.7	1.9	77
PI-40-H	1.10	1.9	0.50	16	60	24.4	2.1	3.7	44.7	3.1	90
DW-40-H	1.07	2.9	0.47	13	80	26.0	2.2	3.5	46.4	3.1	100
TA-40-H	1.23	12.2	0.89	17	115	32.3	2.1	5.0	54.6	3.3	91
FG-40-H	1.24	9.6	0.41	17	90	35.4	2.0	5.1	58.6	3.5	111
CC-40-H	1.24	15.1	1.34	19	470	21.8	3.8	2.2	39.8	1.8	57
PP-00-H	0.90	16.5	1.91	17	540	30.9	9.7	1.4	40.7	1.2	55
Average COV (%)				11	11	1	6	7	1	3	3

[a] Melt index measurements taken at 190°C

Table 4—Summary of properties and performance: Copolymer

Formulation	Specific gravity	Melt index[a] (g/10 min)	Mold shrinkage (%)	Izod impact (J/m) Notched	Izod impact (J/m) Unnotched	Tensile properties Strength (MPa)	Tensile properties Elongation (%)	Tensile properties Modulus (GPa)	Flexural properties Strength (MPa)	Flexural properties Modulus (GPa)	Heat deflection (°C)
PI-20-C	0.98	3.5	0.99	80	160	14.5	3.8	1.5	23.9	1.0	58
DW-20-C	0.97	2.9	0.96	77	180	14.1	3.6	1.4	22.6	1.0	60
PI-40-C	1.08	0.5	0.50	45	75	15.0	2.0	2.9	26.5	1.9	69
DW-40-C	1.07	0.5	0.32	37	75	17.2	1.3	3.2	28.9	2.3	80
TA-40-C	1.25	1.8	0.59	78	430	16.9	2.6	2.3	27.1	2.0	63
FG-40-C	1.22	4.5	0.40	54	140	23.8	3.5	3.7	37.3	2.2	85
CC-40-C	1.19	7.3	1.61	458	745	13.3	2.3	1.4	21.8	0.9	52
PP-00-C	0.90	7.4	2.02	675	730	14.6	9.1	0.7	18.2	0.5	47
Average COV (%)				8	10	2	7	8	2	3	2

[a] Melt index measurements taken at 190°C.

DISCUSSION

Our discussion is divided into three sections. The first discusses SEM investigation. The second section comments on the results as presented in Tables 3 and 4. The third section discusses notched impact, flexural modulus, and maximum tensile strength when specific gravity is taken into account.

SEM Discussion

Figures 1-6 are representative micrographs of the SEM investigations. Photographs of fracture surfaces of the various composites as well as fibers extracted from the fiberglass are shown. Various magnifications were used for the different fillers in order to adequately show the microstructure of the composites. No visual difference was found between the filled homopolymer and filled copolymer, so only homopolymer micrographs are included.

Several general observations were made. First, the fracture surfaces show void-free composites with well dispersed and distributed fillers and good contact between filler and matrix. Second, the varying nature of the fillers represented in this study are readily apparent. For instance, it is easy to see the difference between wood flour and demolition wood fiber. Third, glass fiber length of approximately 0.1 to 1.0 mm can be seen, indicating good length retention after compounding and injection molding

General Discussion

Specific Gravity –Specific gravity values reported agree with other sources (1,3). The slightly lower specific gravity of demolition wood fiber compared with wood flour may be due to minor processing losses incurred while handlimg this relatively low bulk density material.

Melt Index –As mentioned previously, to keep the wood fillers from degrading, melt index (MI) measurements were taken at 190°C instead of 230°C. The lower temperature resulted in a significant decrease of the measured MI for the unfilled polymers, indicating that MI measurements taken at this low temperature should be used for comparative purposes only.

The formulations containing calcium carbonate had the highest MI of the filled formulations; this was probably helped by the stearic acid added by the supplier. As might be expected, the 20% wood-filled formulations had higher readings than did the 40% wood-filled formulations. The 20% wood-filled

formulations were similar to fiberglass, suggesting that fibrous materials at relatively equal volume fractions may have similar MI values. Our experience suggests that wood-filled formulations actually injection mold much better than their MI values taken at 190°C would indicate. Viscosity data at shear rates typical of injection molding would be useful to understand this observation.

Mold Shrinkage—Mold shrinkage values reported for the minerals agree with published sources (3). As a group, all the 20% wood-tilled specimens had similar shrinkage, as did all the 40% wood-filled specimens.

Impact Performance –Calcium carbonate is known for its excellent impact performance, and the numbers indicate its overall superior performance. The pine wood flour and demolition wood had similar performance at either 20% or 40% loadings.

The notched copolymer data show that the 20% wood filled and the talc had similar performance. Unnotched copolymer data for talc were superior to all the wood fillers. The relatively poor performance of fiberglass in the copolymer can reasonably be attributed to a lack of a coupling agent. Statistical variability in the notched homopolymer data precludes definitive discussion.

Tensile Properties—For all practical purposes, the pine wood flour and demolition wood performed about the same, with elongation decreasing and modulus increasing with fiber content. As a class, all the wood fillers outperformed the calcium carbonate, but fiberglass had better overall performance. In the homopolymer, talc performed better than any of the wood fillers, but not quite as good as fiberglass. In the copolymer, talc performed about in the middle of the range described by the wood fillers.

Flexural Properties—Strength values for demolition wood and pine wood flour were about the same, with modulus increasing with filler amount. All the wood-filled formulations were nearly equal or better than calcium carbonate in respect to strength and modulus. Talc and fiberglass were somewhat stronger than the wood-filled formulations, but the 40% wood-filled formulations had similar modulus.

Heat Deflection Temperatures—Fiberglass had the highest heat deflection temperatures, with the 40% demolition wood formulations a close second. Talc performed about the same as pine wood flour and demolition wood at 40% in the homopolymer, but in the copolymer, only talc performed about the same as pine wood flour and demolition wood at 20%. Pine wood flour and demolition wood performed better than calcium carbonate regardless of the loading.

Specific Performance

Differences in mineral and wood fillers become more apparent when they are compared on the basis of their specific gravity. This is important when part weight is critical to overall product performance. The properties previously discussed were derived by testing samples of a given voiume, but differing densities. Testing of equal weight samples is impractical, so discussion in this section is based on results of dividing the property value of a given formulation by its specific gravity. This was done for tensile strength, flexural MOE, and notched Izod impact (Table 5 for homopolymer. Table 6 for copolymer). Figures 7-12 show the relative performance for both test and specific performance values.

Table 5—Summary of specific properties (value/specific gravity): Homopolymer

Formulation	Specific notched Izod (J/m)	Specific flexural MOE (GPa)	Specific tensile strength (MPa)
PI-20-H	13	1.7	26.4
DW-20-H	12	1.9	27.0
PI-40-H	15	2.8	22.2
DW-40-H	12	2.8	24.3
TA-40-H	14	2.7	26.3
FG-40-H	14	2.8	28.6
CC-40-H	15	1.4	17.6

Table 6—Summary of specific properties (value/specific gravity): Copolymer

Formulation	Specific notched Izod (J/m)	Specific flexural MOE (GPa)	Specific tensile strength (MPa)
PI-20-C	84	1.1	14.8
DW-20-C	79	1.1	14.5
PI-40-C	41	1.8	13.9
DW-40-C	35	2.2	16.1
TA-40-C	62	1.6	13.5
FG-40-C	45	1.8	19.5
CC-40-C	385	0.8	11.2

Notched Izod (Specific performance)—For the homopolymer, the relative performance of the fillers could be considered equal. For the copolymer, calcium carbonate had the best performance, followed by the 20% wood-loaded formulations. Talc performed about midway between the range described by the wood fillers. The poor performance of fiberglass is likely attributable to the lack of coupling agent.

Flexural MOE (Specific Performance)—In the homopolymer, 40% loaded demolition wood and pine wood flour had nearly identical performance to fiberglass and talc. The 20% loaded demolition wood and pine wood flour had similar performance to each other, followed by calcium carbonate.

For the copolymer, the 40% demolition wood had better performance than talc or fiberglass. The 40% loaded pine wood flour, fiberglass, and talc had equivalent performance. Calcium carbonate had the lowest performance of any fillers in this category.

Maximum Tensile Strength (Specific Performance)–For the homopolymer, fiberglass, talc, pine wood flour, and demolition wood at 20% had nearly equivalent performance, perhaps as a result of approximately the same polymer displacement. Pine wood flour and demolition wood at 40% loadings performed better than calcium carbonate, which had the poorest performance in this class.

For the copolymer, fiberglass had the best performance, followed by demolition wood at 40%. The performance of talc, pine wood flour at 40% and 20% , and demolition wood at 20% was about the same. Caicium carbonate had the lowest performance.

CONCLUSIONS

We conducted this study to determine some baseline comparative data between several wood fillers and mineral and glass fillers in injection molding grade polypropylene. Because it was not our purpose to optimize any particular performance aspect of the fillers, no additional coupling agents, compatabilizers, or impact modifiers were included in the study.

For the fillers and polymer used in this study, we conclude the following:

- Fiberglass is a useful filler for applications needing high levels of modulus of elasticity (MOE) and maximum tensile strength (MTS), where cost and specific gravity are of minimal concern.
- Calcium carbonate is a useful filler for impact properties, but gains in MOE and MTS were minimal.
- For the homopolymer, wood fillers had similar performance to talc, with lower specific gravity.
- For the copolymer polypropylene, wood fillers also had similar performance to talc with lower specific gravity, with the exception of unnotched Izod impact performance.
- The data do not substantiate a consistent relationship between equivalent volume percentage loadings (20% wood-filled formulations compared with 40% mineral- or glass-filled formulations).

REFERENCES

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2. ASTM. 1990. Annual Book of ASTM Standards, Vol. 8. Philadelphia, PA: American Society of Testing and Materials.

3. Modern Plastics Encyclopedia. 1989. New York: McGraw Hill.

Figure 1—Fracture surface of 40% pine wood flour (x30).

Figure 2—Fracture surface of 40% talc (x1,000).

Figure 3—Fracture surface of 40% calcium carbonate (x1,000).

Figure 4—Fracture surface of 40% demoliton wood (x220).

Figure 5—Fracture surface of 40% fiberglass (x120).

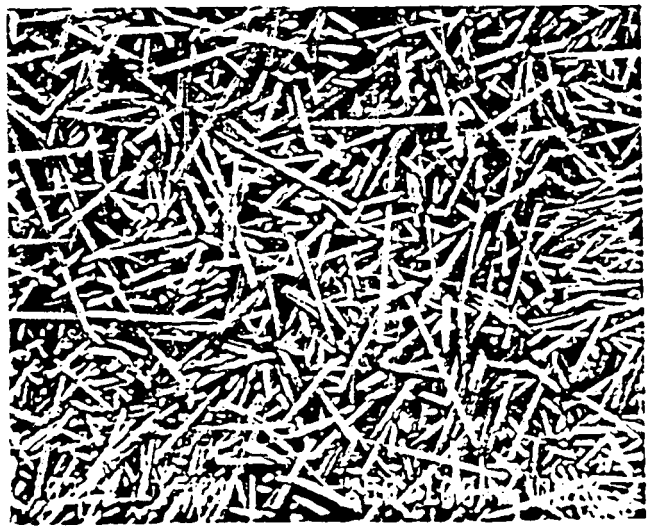

Figure 6—Extracted fiberglass (x50).

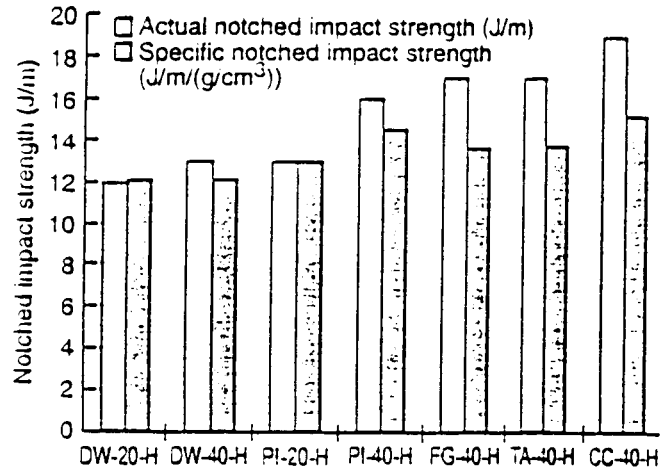

Figure 7—Notched impact strength: Homopolymer

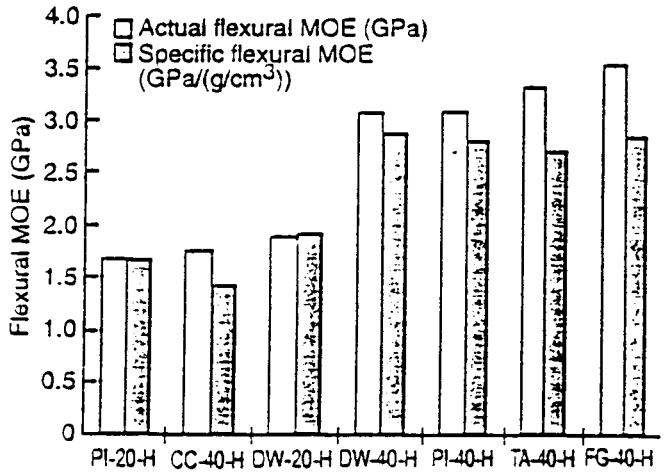

Figure 8—Flexural MOE: Homopolymer

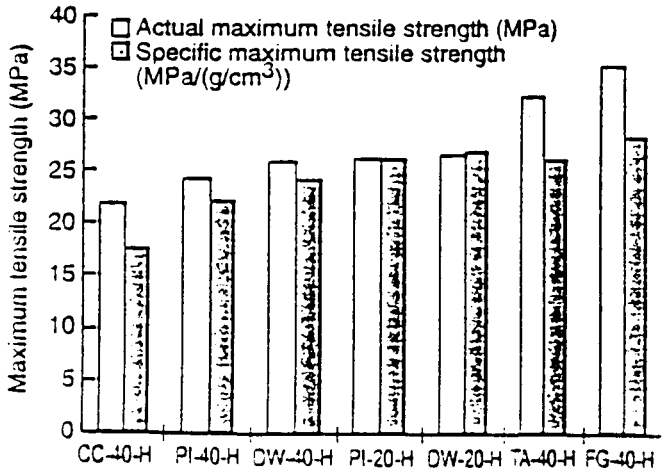

Figure 9—Maximum tensile strength: Homopolymer

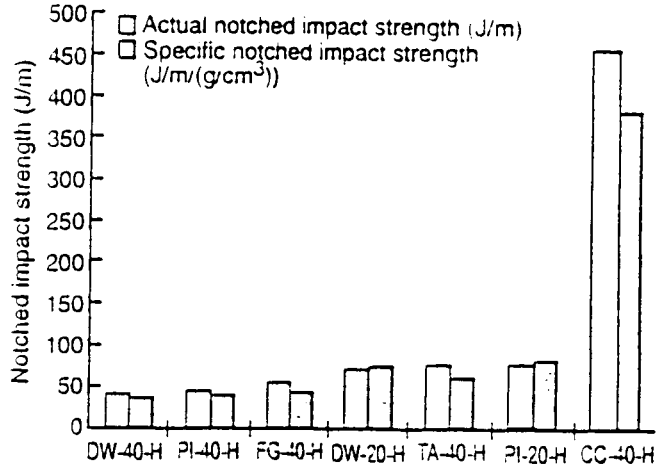

Figure 10—Notched impact strength: Copolymer

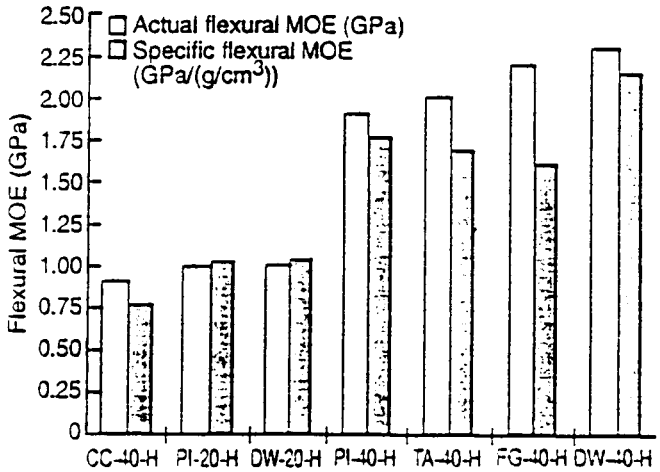

Figure 11—Flexural MOE: Copolymer

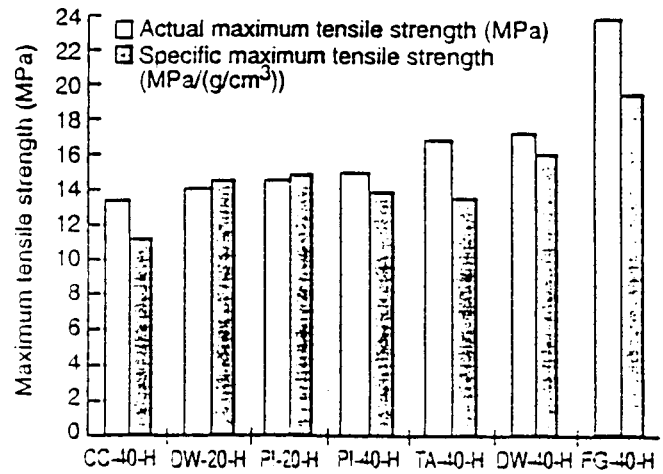

Figure 12—Maximum tensile strength: Copolymer