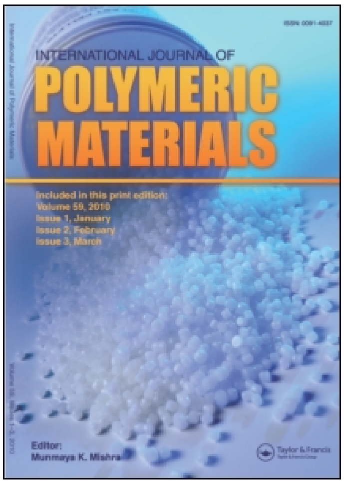

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MATHEMATICAL MODELING AND THE ESTIMATION OF PARAMETERS FOR THE MELT VISCOSITY OF POLYAMIDES

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Capillary rheometry data of a range of commercial polyamide engineering materials was obtained from a mould-flow analysis material database, from which melt viscosity data was obtained at different temperatures, which made comparison of the viscosities difficult. In an attempt to make a reasonable comparison between the melt viscosities of the various polyamide materials at different temperatures, it was necessary to obtain the mathematical functions which describe the relationships between: (i) the melt viscosity and the shear rate, (ii) the melt viscosity and temperature and (iii) the melt viscosity and the combined effect of the shear rate and temperature of each of the polyamides studied. Therefore, melt viscosity was modelled as a function of shear rate at the three temperatures (275°C, 295°C and 315°C), at which the viscosities were determined. The function obtained represented smoothed versions of the experimental data, eliminating the experimental noise and enabling the generation of melt viscosity data at the six different shear rates of the original data. It was established that the melt viscosity as a function of shear rate at constant temperature, in the shear rate range 500–700 s^{-1}, is incorrectly described by the Ostwald-de-Waele's model, $\eta_T = f(\dot{\gamma}) = K(\dot{\gamma})^{(n-1)}$*, while the melt viscosity of the polyamides studied, as a function of temperature, is correctly described by the model* $\eta_{\dot{\gamma}} = g(T) = Pe^{(QT/R)}$*. But the response-surface melt viscosity is effectively described as a function of both shear rate and temperature by the model:* $\eta = |\dot{\gamma}|^{(n-1)} Ae^{(ET/R)}$*. The parameters A, E and n are highly interrelated as they all influence the average melt viscosity. All are temperature sensitive and also, to some degree, shear sensitive.*

Keywords: melt viscosity, shear rate, shear stress, shear flow, temperature, location parameter, melt consistency index, shear rate factor

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INTRODUCTION

Polyamide engineering plastics comprise a family of related materials that differ in terms of chemical structure and various types of additives and fillers [1]. Typical properties of polyamide 6 and polyamide 66 are their toughness over a wide range of temperatures, impact and abrasion resistance, lubricity and good resistance to organic solvents and petroleum products [2–13]. The science of the development of polyamide engineering plastic formulations is driven by the needs of the injection moulding market, requesting that the materials be tailored to specific end-use requirements [2, 3, 14]. These are usually expressed in terms of their physical and mechanical properties, cost and ease of injection moulding (processability). Polyamide compounding can be defined as the combination of a base polyamide polymer with colorants, modifiers, additives, reinforcements, fillers or other polymers, to make the base polymer perform better, cost less, process more easily, be aesthetically attractive or otherwise improve its characteristics [15]. By copolymerization, reinforcement with glass, toughening with rubber and control of molecular mass, a wide range of mechanical properties can be achieved [16]. Heat stabilizers and other modifiers increase long-term oxidative and hydrolytic stability. In other words, through compounding, polyamides can be modified to meet any one (or more) of hundreds of different performance/processing/price parameters, dictated by the requirements of the supplier, processor or end-user.

Every time, compound development is frequently and continuously carried out in an empirical manner. However, problems are not systematically treated and solutions are seldom based on the knowledge of material properties. Problems are usually solved with the focus only on the particular problem in hand at that particular time, the technical equipment available and knowledge of the requirements which plastic articles have to satisfy [17]. The effects of variations in polymer grade, polymer type, additive type and level in a particular plastic, are usually determined and interpreted in isolated studies. They are described in terms of the physical properties of injection moulded test pieces, and not in terms of an important material property such as melt viscosity or in terms of processability. Knowledge of the effects of the composition of the material being processed on the melt flow properties of polyamide engineering compounds is a fundamental prerequisite to providing the preconditions for selective development and to select the compounding conditions in order to achieve the desired properties [17]. This knowledge is invaluable in ensuring that compounded polyamide products are consistent in terms of both the physical properties and the injection moulding processability. Furthermore, it enables the formulator to predict the injection moulding behaviour of developed compounds.

Most often, published information on the relationships between the composition and the melt flow behaviour of polyamide engineering compounds gives qualitative descriptions of different polyamide/filler/melt viscosity interactions. Such descriptions are usually not supplied under comparable conditions of temperature and shear, hence this does not permit ready and accurate comparisons of the melt viscosities of different types of polyamide containing different types and/or levels of fillers and other additives. Studies [18–24] of the melt flow properties of polyamide compounds by capillary rheometry have been reported. Accurate experimental data of commercial polyamide engineering plastic compounds commonly used for mould flow analysis, as determined in accordance with the internationally recognized standards (ISO), are available in the international plastics databases [21], albeit in a format not permitting functional comparisons.

It should be noted that although the melt viscosity at any given shear rate and temperature is a function of the zero viscosity of the polyamide compound, extrapolation from the shear rate range studied to the zero viscosity is not recommended. While zero viscosity of a polymer melt increases with increasing mean molecular mass, the viscosity drop in the transition from Newtonian to pseudoplastic behaviour correlates with the width of the molecular mass distribution [19]. Generally, the broadening of the molecular mass distribution produces increasing shear sensitivity [14]. Measurements of melt viscosity at low shear rates should not be used to predict injection moulding processes at high shear rates, because viscosity versus shear rate curves do diverge, converge or even cross, giving very poor predictability [25]. Yucel and Ozddogan [26] suggested a semi-theoretical method based on the Enskog's hard sphere theory for dense fluids and the principle of corresponding states for predicting the viscosities of pure organic liquids. The method, however requires only normal boiling point and critical property data to predict the viscosity of a pure liquid at any given temperature between $0.45 < T_r < 0.80$, where T_r is the reduced temperature, T/T_c (and T is the absolute temperature while T_c is the critical temperature). Non-Newtonian flow behaviour was successfully modelled by De Kee *et al*. [27] with a simplified equation derived from a model proposed by De Kee and Chan Man Fong [28]. Yield stress and viscosity data were found to obey the Arrhenius-type relation over the temperature range investigated.

The purpose of this study, therefore, is to investigate the dependence of melt viscosity on temperature and shear rate, so as to further understand the melt flow behaviour of polyamides. This knowledge is needed for state-of-the art compounding, since it is not generally accessible to custom compounders and is viewed as proprietary by major polyamide manufacturers. It is therefore necessary to:

(a) acquire the "equivalent" of the inaccessible proprietary information from available melt-viscosity data, determined by capillary rheometry in accordance to the ISO, through the utilization of generally available processing techniques,
(b) test the validity and applicability of known melt-viscosity equations which relate melt viscosity to melt temperature and/or shear rate and
(c) mathematically, describe the relationships between the composition and melt-flow properties of different polyamide injection moulding compounds by using appropriate equations and modeling non-linear parameters as functions of the temperatures and shear rates characteristic of injection moulding processes.

THEORETICAL BACKGROUND

Effect of Temperature and Pressure on the Melt Viscosity

It is generally recognized that increasing temperature increases atomic vibration and molecular mobility, thus reducing melt viscosity [22]. The variation of viscosity with both temperature and pressure is a manifestation of its dependence on a more fundamental quantity, the free volume. Free volume is the volume of the space in a melt that is not actually occupied by molecules and is thus available to permit the mobility of the molecules. The greater the free volume, the easier it will be for the molecules to adjust to deformations and this will be reflected in a lower viscosity. Increasing temperature results in thermal expansion and thus an increase in the free volume. This explains the decrease in viscosity as the temperature increases. Increasing the pressure, on the other hand, results in compression, and thus a decrease in free volume and an increase in viscosity. Since polymers are not very compressible, the dependence of free volume on pressure, and thus on viscosity, is not nearly as important as its dependence on temperature. For polymers that exhibit Newtonian type of flow, the Arrhenius-type equation (Eq. 1) is often used to express melt viscosity as a function of temperature [20]. With such an equation, the highly viscous polymers will, no doubt, be more sensitive to the effects of temperature.

$$\eta(\mathrm{T}) = \mathrm{Ae}^{(\mathrm{E_a T/R})} \tag{1}$$

where A is the viscosity factor, E_a is the energy of activation, R the gas constant and T the absolute temperature.

Another equation often used to describe the temperature dependence of viscosity [25] is given thus:

$$\eta_2(\mathrm{T_2}) = \eta_1(\mathrm{T_1}) - \mathrm{E}\left(\frac{\mathrm{T_2} - \mathrm{T_1}}{\mathrm{T_1}}\right) \tag{2}$$

where η_1 and η_2 are melt viscosities at temperatures T_1 and T_2 respectively and E is the temperature coefficient. This suggests that if the logarithm of viscosity is plotted as a function of temperature, a straight line with a slope of $-$E will result. Equation (2) gives a good approximation over a temperature range of between 275°C and 315°C.

The Effect of Molecular Mass on the Melt Viscosity

Plastics require large molecule to provide the cohesion for most end-use properties. Thermoplastic injection moulding requires a 'compromise' molecular mass, low enough for reasonably easy processing, but high enough to ensure reasonably good end-use properties. It is generally accepted that increasing molecular mass produces increasing melt viscosity hence making the polymer to be more difficult for injection moulding. This relationship is quantified by the equation [25]:

$$\eta_{\mathrm{melt}} = \beta \mathrm{M}_w^{\alpha} \tag{3}$$

where η_{melt} is the melt viscosity, β is the proportionality constant, M_w is the weight-average molecular mass and α is the molecular mass factor. The proportionality constant depends on the flexibility and intermolecular attraction of the polymer molecules and on processing conditions, *i.e.*, temperature, pressure and shear rate [22]. Equation (3) applies when a critical value of molecular mass, associated with the onset of chain entanglement, is exceeded [16, 20]. Increasing the molecular mass not only increases melt viscosity, but also rubbery melt-flow. The latter is due to the inability of molecules to disentangle completely within the limited temperature, pressure, shear rate and time span of the process. An elastic melt can result in a variety of injection moulding problems, such as post-mould shrinkage, warping and cracking [20].

Effect of Molecular Mass Distribution on the Melt Viscosity

While the zero viscosity of polymer melts increases with increasing mean molecular mass, the viscosity drop in the transition from Newtonian to pseudoplastic behaviour correlates with the width of the molecular mass distribution [19].

Shear Sensitivity Effect on Melt Viscosity

At low shear rates, shear stress is directly proportional to the shear rate and therefore the viscosity is independent of the shear rate. This is Newtonian melt flow behaviour. Viscosity is a maximum here and is described as the zero viscosity. This Newtonian range is followed by a non-Newtonian range at higher shear rates, where polymer melts become less viscous at increasing shear rates, probably because of the partial orientation of the molecules during unidirectional flow [20, 22–24]. This is the range of shear rates through which the melts have to pass during the various processing methods. Accordingly, viscosity in this range decreases with increasing shear rate. This is the pseudoplastic melt flow behaviour, meaning that the output of an extruder, for instance, increases non-proportionately with increasing shear rate. Very few polymers exhibit the reverse trend of increasing the viscosity associated with dilatant materials [29, 30].

EXPERIMENTAL

Mathematical Modeling and Estimation of Parameters

Capillary rheometry data of a range of commercial polyamide engineering thermoplastics (27 in total) was obtained from a mould-flow analysis, material-data base [9] (see Appendix). Because the melt viscosities of the various polyamide grades were determined at different temperatures, ready comparison of these materials was impossible (or at best difficult), hence the need for the modeling and estimation of parameters necessary to predict the melt behaviours of these materials.

To facilitate comparisons of the melt viscosities of these materials, it was necessary to obtain the mathematical functions which best describe the melt viscosity/shear rate/temperature relationships of each of the polyamides studied. To achieve this aim, therefore:

(1) melt viscosity was modelled as a function of shear rate at each of the three temperatures (275°C, 295°C and 315°C) at which the melt viscosities had been measured. The functions obtained represented

smoothed versions of the experimental data, thereby removing the experimental noise, following which it was possible to generate the melt viscosity values at the six different shear rates (500 s^{-1}, 700 s^{-1}, 1000 s^{-1}, 2000 s^{-1}, 5000 s^{-1} and 7000 s^{-1}) of the original data. (2) melt viscosity as a function of temperature was modelled from the generated data set at each of the shear rates. (3) the models for polyamide melt viscosity as a function of shear rate and as a function of temperature were combined, allowing the estimation of parameters of the three-dimensional model for each of the polyamide grades.

METHODS

Modeling of Melt Viscosity as a Function of Shear Rate at Constant Temperature

The shear rate dependence of melt viscosity at constant temperature is described by the function:

$$\eta_T = f(\dot{\gamma}) = K(\dot{\gamma})^{(n-1)} \tag{4}$$

where η_T is the melt viscosity at temperature T, $\dot{\gamma}$ is the shear rate and K, the melt consistency index and n, the shear rate factor are the parameters to be estimated.

The non-linear estimation was performed with SYSTAT's non-linear software [27]. The non-linear least-squares loss-function was employed together with the Simplex estimation method. This method allows estimations of the deviations of the dependent variable from values estimated from the independent variable data points, which were squared and iteratively minimized. Parameters were estimated for melt viscosity as a function of shear rate at constant melt temperatures. Three sets of parameters were estimated at the three chosen temperatures, for each of the 27-polyamide grades studied.

Calculation of Melt Viscosity Using Estimated Parameters, A and E

Equation (3) and parameter set {A, E} were employed to calculate melt viscosity values at six different shear rates. To correlate the estimated melt viscosity data with melt temperatures at constant shear rates, the following function was selected:

$$\eta_{\dot{\gamma}} = g(T) = Pe^{(QT/R)} \tag{5}$$

P, the viscosity factor and Q, the temperature coefficient are the estimated parameters, similar to the Arrhenius constant and activation energy, respectively, in Eq. (1), T is the temperature, R, is the gas constant and $\eta_{\dot{\gamma}}$ is the viscosity dependence on temperature at constant shear rate. Non-linear estimation was performed with non-linear software. As in method 1 above, the non-linear least-squares loss-function was used together with the Simplex estimation method. Parameters were estimated for melt viscosity as a function of melt temperature at constant shear rates. Six sets of parameters were estimated at the chosen six different shear rates, for the 27-polyamide grades studied.

Modeling of Melt Viscosity as a Function of Both the Melt Temperature and the Shear Rate

To determine the dependence of melt viscosity, η, on both the melt temperature and shear rate, Eqs. (4) and (5) were combined to yield:

$$\eta = |\dot{\gamma}|^{(\mathrm{n}-1)}\mathrm{A}\mathrm{e}^{(\mathrm{ET/R})} \qquad (6)$$

where A is the viscosity factor, E is the temperature coefficient and n is the shear rate factor. Equation (6) was used to model melt viscosity as a function of both shear rate and temperature. Again, non-linear estimation was performed with SYSTAT's non-linear software and non-linear least-squares loss-function was used with the Simplex estimation method. Parameters A, E and n were estimated for each of the polymer grades studied.

RESULTS AND DISCUSSION

Dependence of Melt Viscosity on Shear Rate at Constant Temperature

Figure 1 shows a schematic diagram of the flow and viscosity curves for a range of shear rates of some practical manufacturing processes. The flow or viscosity curves of most polymer melts are similar to the curves in Figure 1, however, the relative degree of shear sensitivity varies greatly from one polymer to another. The non-linear behaviour is attributed to the gradual transition of high molecular fractions to rubbery (non-fluid) state [22]. Chart 1 shows, pictorially, the routes taken for the modeling of melt viscosity. While Chart 1 depicts an overview of the routes taken for the modeling of melt viscosity as a function of shear rate at constant temperature to generate or estimate the parameters K and n, Table 1 shows the estimated parameters with

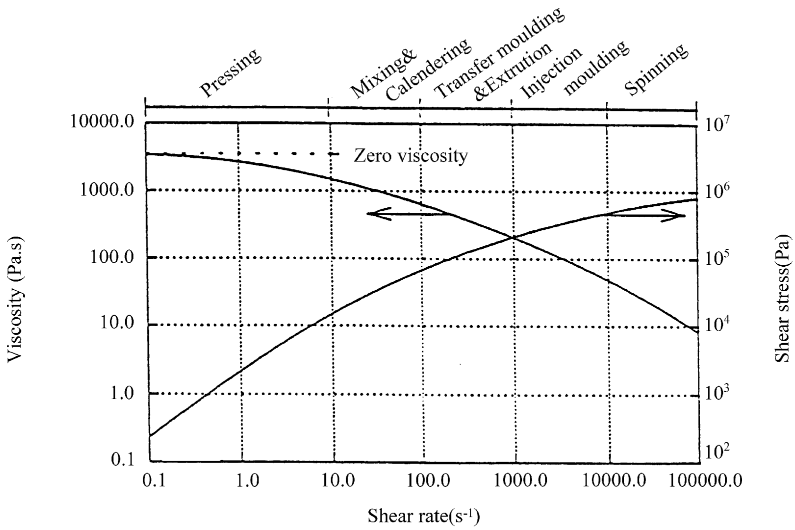


FIGURE 1 A schematic diagram of flow and viscosity curves for a shear rate range of some practical manufacturing processes.

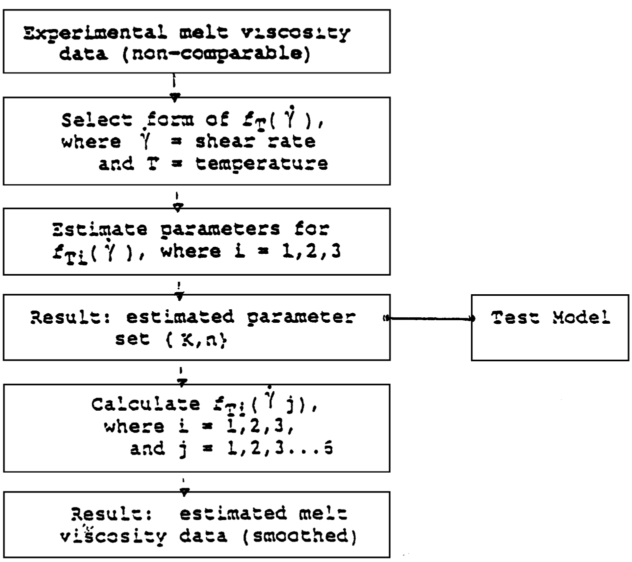


CHART 1 A schematic overview of the modeling of melts viscosity as a function of shear rate at constant temperature.

TABLE 1 Estimated Parameters K and n and Melt Viscosities Calculated at Different Shear Rates, $\dot{\gamma}$ and Temperatures with Eq. (4). Results are for some of the Resins

Resin grade no.	Modeling temp., °C	K	n	Shear rate, 500 s^{-1}	Shear rate, 700 s^{-1}	Shear rate, 1000 s^{-1}	Shear rate, 2000 s^{-1}	Shear rate, 5000 s^{-1}	Shear rate, 7000 s^{-1}	r^2	r^2 corrected
16	275	3411.282	0.587	262	228	197	148	101	88	1.000	1.000
16	290	2063.076	0.594	165	144	125	94	65	57	1.000	1.000
16	305	1258.301	0.602	106	93	80	61	42	37	1.000	1.000
17	275	2974.504	0.586	227	197	170	128	88	76	1.000	0.999
17	290	1786.593	0.603	152	133	115	87	61	53	1.000	.0999
17	305	1070.969	0.623	103	91	79	61	43	38	1.000	0.998
18	275	4831.511	0.539	275	236	200	145	95	82	1.000	1.000
18	290	2844.954	0.546	169	145	124	90	60	51	1.000	1.000
18	305	1712.129	0.553	106	92	78	57	38	33	1.000	1.000
19	275	3644.789	0.523	188	160	135	97	6353	1.000	1.000	
19	290	2274.056	0.515	112	95	80	57	37	31	1.000	1.000
19	305	1385.078	0.515	68	58	49	35	22	19	1.000	1.000
20	275	4509.310	0.560	293	253	216	159	106	92	1.000	1.000
20	290	2769.061	0.558	178	153	131	96	64	55	1.000	1.000
20	305	1708.175	0.559	110	95	81	60	40	34	1.000	1.000
21	275	4096.105	0.544	241	207	176	128	84	72	1.000	0.999
21	290	2851.090	0.551	175	151	128	94	62	54	1.000	0.999
21	305	2023.644	0.558	130	112	96	70	47	40	1.000	0.999

22	275	854.552	0.696	129	117	105	85	64	58	0.998	0.977
22	290	349.121	0.761	79	73	67	57	46	42	0.998	0.967
22	305	152.480	0.816	49	46	43	38	32	30	0.999	0.957
23	275	1536.215	0.622	147	129	113	87	61	54	0.999	0.994
23	290	767.063	0.670	99	88	78	62	46	41	0.999	0.990
23	305	387.186	0.715	66	60	54	44	34	31	0.999	0.986
24	275	42063.183	0.365	813	657	523	337	188	152	1.000	0.999
24	290	22763.351	0.416	604	496	403	269	157	129	1.000	0.998
24	305	16332.652	0.443	513	425	348	237	142	118	0.999	0.997
25	275	1719.398	0.629	171	151	133	102	73	64	1.000	1.000
25	290	985.130	0.646	109	97	85	67	48	43	1.000	0.999
25	305	559.922	0.664	69	62	55	44	32	29	1.000	1.000
26	275	3804.381	0.565	255	220	188	139	94	81	1.000	0.998
26	290	1944.990	0.603	165	144	125	95	66	58	0.999	0.996
26	305	956.484	0.647	107	95	83	65	47	42	0.999	0.994
27	275	5944.754	0.535	330	283	239	173	113	97	1.000	1.000
27	290	3628.698	0.552	224	193	164	120	80	69	1.000	1.000
27	305	2184.878	0.571	152	131	113	84	57	49	1.000	0.999
28	275	904.133	0.699	139	126	113	92	70	63	1.000	0.997
28	290	499.090	0.743	101	93	85	71	56	51	1.000	0.995
28	305	272.077	0.784	71	66	61	53	43	40	1.000	0.994
29	275	2974.504	0.586	227	197	170	128	88	76	1.000	0.999
29	290	1786.593	0.603	152	133	115	87	61	53	1.000	0.999
29	305	1070.969	0.623	103	91	79	61	38	38	1.000	0.998

K = melt consistency index, n = shear rate factor and r^2 = coefficient of fit.

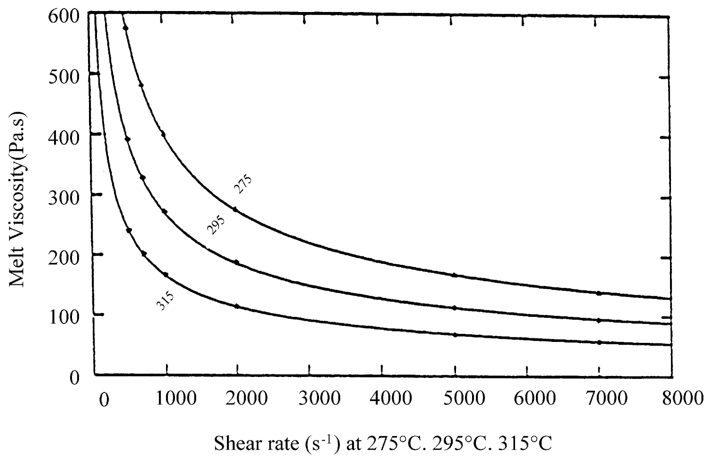


FIGURE 2 Melt viscosity as a function of shear rate at different temperatures.

the regression coefficients. Parameters K and n are significant and the validity of the model is confirmed with strong regression coefficients. Figure 2 shows melt viscosity as a function of shear rate at the chosen three different temperatures. Six plots out of the 27 studied for the dependence of melt viscosity on shear rate are shown in Figure 3. Figure 4 shows melt viscosity as a function of the melt temperatures

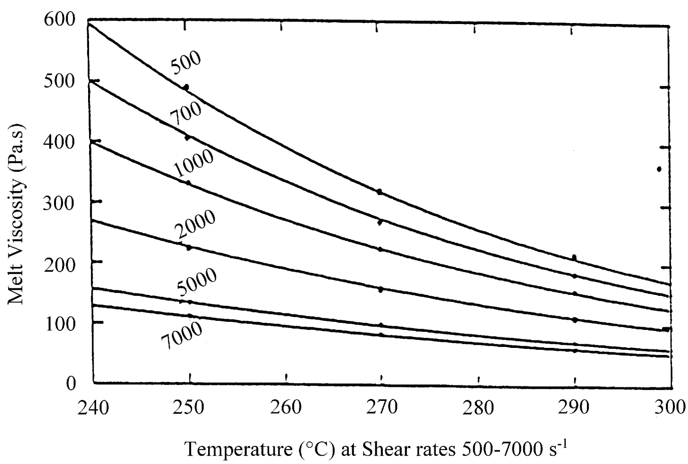


FIGURE 3 Melt viscosity as a function of temperature at different shear rates.

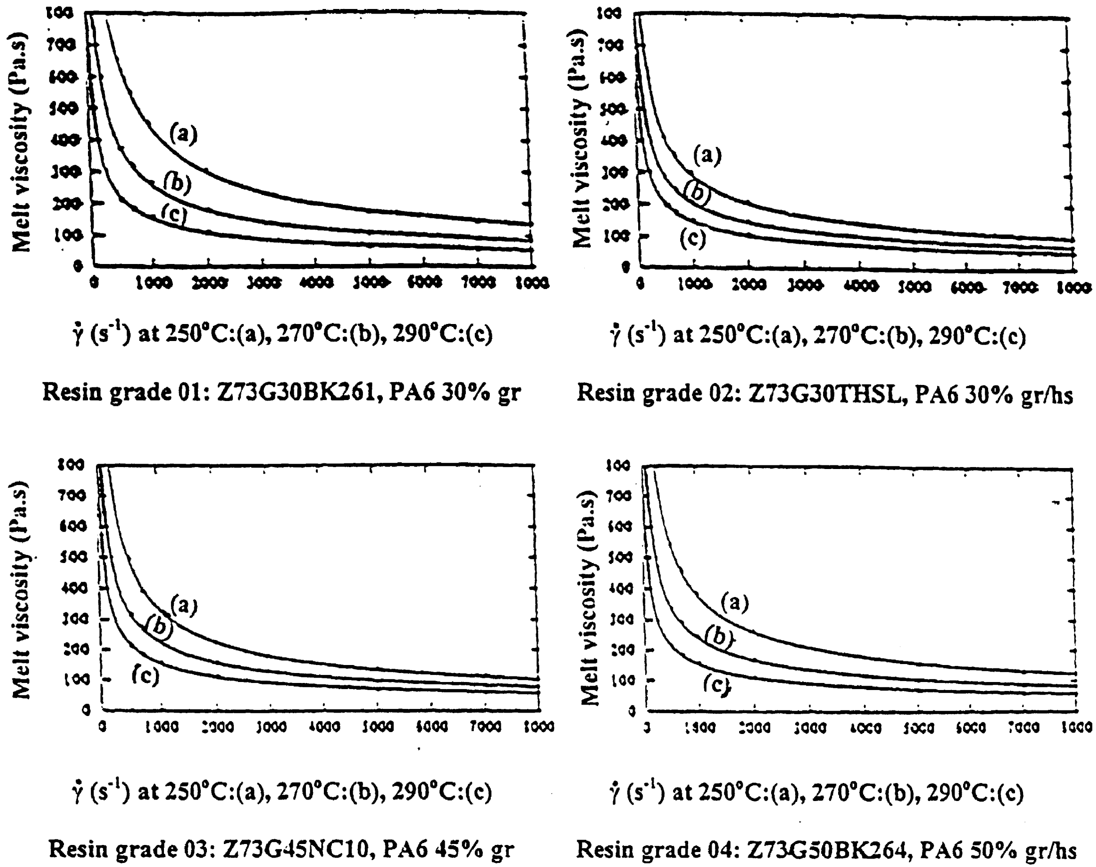
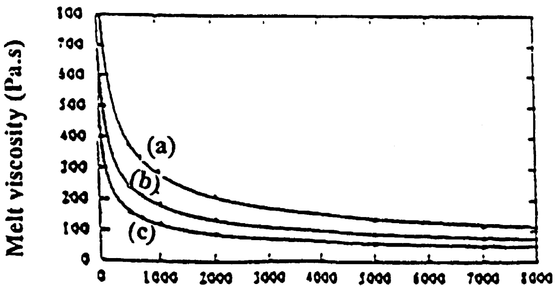
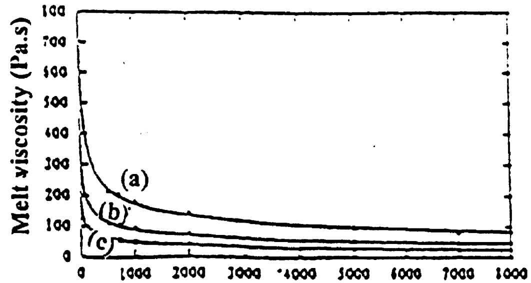


FIGURE 4 Modeling melt viscosity as a function of shear rate at constant temperatures for a few selected samples of polyamides. (*Continued*)



$\dot{\gamma}$ (s^{-1}) at 250°C:(a), 270°C:(b), 290°C:(c)

Resin grade 05: M13T2GY282,
PA66 30% mr/im



$\dot{\gamma}$ (s^{-1}) at 250°C:(a), 270°C:(b), 290°C:(c)

Resin grade 06: M10B140NC10, PA66 40% mr

FIGURE 4 Continued.

for the chosen six shear rates. The others show similar trends. At low shear rates (500–1000 s^{-1}) the melt viscosity decreases almost non-linearly with increasing shear rate, levelling-off at higher shear rates. As the melt temperature increases, the slope of the function becomes smaller, indicating decreasing shear sensitivity with increasing temperature. For a given polyamide grade at a given melt temperature, the parameter K is proportional to the magnitude of the melt viscosity at the lower end of the shear rate range. The parameter n describes the rate of the melt viscosity decrease as a function of increasing shear rate. The smaller the numerical value of n, the greater the shear sensitivity of the polyamide material at that temperature. The model correctly describes the melt viscosity of polyamide compounds as a function of shear rates, $\dot{\gamma} > 700\,s^{-1}$, at constant temperature.

Dependence of Melt Viscosity on Temperature at Constant Shear Rate

Chart 2 gives an overview of the routes followed for the modeling of melt viscosity as a function of temperature at constant shear rate in order to generate the parameters A and E, while the values of these parameters together with the regression coefficients are shown in Table 2. Figure 3, giving the general fit of the function g(T), shows the melt viscosity plot as a function of temperature at different shear rates. Increasing both the melt temperatures and the shear rates

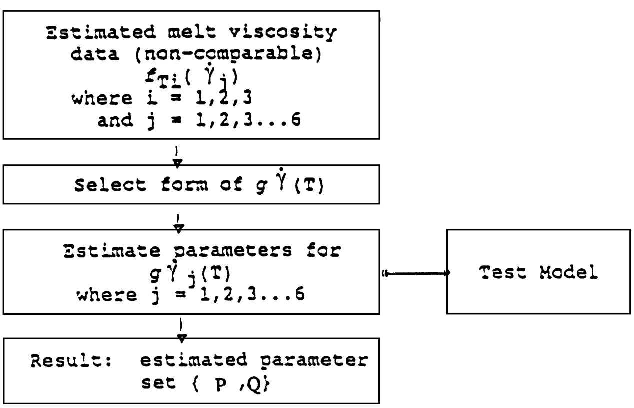


CHART 2 A schematic overview of the modeling of melts viscosity as a function of temperature at constant shear rate.

TABLE 2 Parameters (P and Q), Estimated at Different Shear Rates, $\dot{\gamma}$ with Eq. (5). Results are for some of the Resins

Resin grade no.	Shear rate (s^{-1})	P	Q	r^2	Corrected r^2
01	500	735971.367	– 0.233	1.000	1.000
01	700	525572.654	– 0.228	1.000	1.000
01	1000	357280.376	– 0.222	1.000	1.000
01	2000	182072.409	– 0.213	1.000	1.000
01	5000	71696.090	– 0.200	1.000	1.000
01	7000	48776.114	– 0.193	1.000	1.000
02	500	38732.861	– 0.151	1.000	1.000
02	700	30144.419	– 0.148	1.000	1.000
02	1000	23759.432	– 0.146	1.000	1.000
02	2000	13917.568	– 0.140	1.000	1.000
02	5000	6958.048	– 0.133	1.000	1.000
02	7000	5497.026	– 0.131	1.000	1.000
03	500	87721.342	– 0.173	1.000	1.000
03	700	60290.704	– 0.166	1.000	1.000
03	1000	39244.757	– 0.159	1.000	1.000
03	2000	18249.756	– 0.146	1.000	1.000
03	5000	6842.341	– 0.131	1.000	0.999
03	7000	4587.696	– 0.124	1.000	1.000
04	500	208120.004	– 0.198	1.000	1.000
04	700	154410.899	– 0.194	1.000	1.000
04	1000	112501.875	– 0.190	1.000	1.000
04	2000	56708.997	– 0.179	1.000	1.000
04	5000	24096.284	– 0.167	1.000	1.000
04	7000	18883.809	– 0.165	1.000	1.000
05	500	148202.276	– 0.180	1.000	1.000
05	700	126312.662	– 0.180	1.000	1.000
05	1000	104457.092	– 0.179	1.000	1.000
05	2000	76892.092	– 0.179	1.000	1.000
05	5000	48036.768	– 0.176	1.000	0.999
05	7000	40190.880	– 0.175	1.000	1.000
06	500	1653168.749	– 0.269	1.000	0.999
06	700	1398319.574	– 0.268	1.000	1.000
06	1000	1196245.558	– 0.267	1.000	1.000
06	2000	757431.078	– 0.261	1.000	1.000
06	5000	422153.124	– 0.253	1.000	1.000
06	7000	360177.283	– 0.252	1.000	0.999
07	500	7308227.263	– 0.289	1.000	1.000
07	700	3860842.275	– 0.275	1.000	1.000
07	1000	1917888.285	– 0.260	1.000	1.000
07	2000	507086.093	– 0.230	1.000	1.000
07	5000	92172.555	– 0.192	1.000	0.999
07	7000	48647.991	– 0.178	1.000	0.999

P = the location parameter, Q = slope which relates melt viscosity with temperature, r^2 = coefficient of fit.

results in a decrease in melt viscosities. Figure 4 shows the representative curves of six of the 27 polyamide compounds studied. The shifts of the melt viscosity-temperature curves with increasing shear rates are non-linear. A power law function (Eq. 4) was used to describe the melt viscosity-shear rate relationship at constant temperature. At higher melt temperatures, the different melt viscosities at different shear rates converge, *i.e.*, the differences in melt viscosities as a function of shear rates, become smaller. It was therefore established that for the polyamide compounds studied, melt viscosity as a function of temperature at constant shear rate, is very well described by the model represented in Eq. (5).

Dependence of Melt Viscosity on the Combined Effect of Melt Temperature and Shear Rate

Chart 3 shows a schematic overview of the modeling routes for determining melt viscosity as a function of both temperature and shear rate, represented in Eq. (6). The estimated parameters A, E and n for each of the polyamide grades under investigation, together with the regression coefficients, are summarized in Table 3. The significance of parameter A is illustrated in Figure 5.

The parameter A is a location parameter for the response surface described in Eq. (6). The magnitude of A is proportional to the average melt viscosity of the polyamide grade in question. As the magnitude of

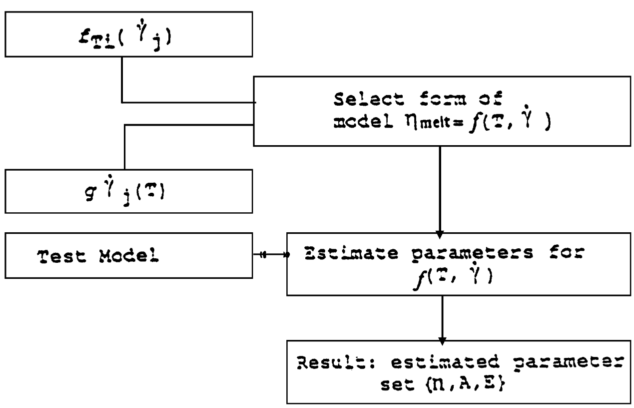


CHART 3 A schematic overview of the modeling of melt viscosity as a function of both shear rate and temperature.

TABLE 3 Parameters A, E, and n Estimated for the Polyamide Resins with Eq. (6). Results are for some of the Resins

Resin grade no.	$A \times 10^4$	E	n	r^2	Corrected r^2
01	1612.160	− 0.226	0.468	0.998	0.994
02	79.915	− 0.147	0.496	1.000	0.999
03	182.520	− 0.163	0.464	0.999	0.997
04	439.066	− 0.191	0.475	1.000	0.999
05	214.425	− 0.180	0.570	1.000	0.998
06	1219.400	− 0.264	0.650	0.999	0.995
07	6010.490	− 0.266	0.532	0.996	0.988
08	3477.350	− 0.324	0.554	1.000	0.999
09	6670.750	− 0.278	0.541	0.999	0.997
10	4.904	− 0.132	0.723	0.998	0.983
11	4.904	− 0.132	0.723	0.998	0.983
12	4.904	− 0.132	0.723	0.998	0.983
13	157.307	− 0.175	0.553	1.000	0.999
14	931.114	− 0.192	0.465	0.998	0.993
15	59.329	− 0.140	0.501	1.000	0.999
16	1281.530	− 0.250	0.591	1.000	1.000
17	311.295	− 0.212	0.595	1.000	0.999
18	2852.130	− 0.263	0.542	1.000	1.000
19	4378.580	− 0.284	0.520	1.000	1.000
20	3770.550	− 0.273	0.559	1.000	1.000
21	109.949	− 0.170	0.548	1.000	0.999
22	215.578	− 0.243	0.727	0.997	0.981
23	105.954	− 0.303	0.648	0.999	0.991
24	200.924	− 0.124	0.397	0.998	0.993
25	612.748	− 0.227	0.585	0.999	0.996
27	589.378	− 0.211	0.545	1.000	0.999
29	311.295	− 0.212	0.595	1.000	0.999

r^2 = coefficient of fit.
Estimated parameters are: A = viscosity factor, E = temperature coefficient and n = shear rate factor.

A increases, while E and n remain constant, the response surface lifts upwards, parallel to the z-axis. Figure 5 shows that with increasing P (*i.e.*, increasing melt viscosity), the response surface becomes both extra temperature and shear rate-sensitive.

Q is the slope, which relates melt viscosity to melt temperature. With increasing magnitude of Q, the overall melt viscosity increases and the response surface becomes more shear sensitive. This is illustrated in Figure 6. The response surface is very sensitive to small changes in the magnitude of parameter Q. The displacement of the response surface corresponds to an 0.02 magnitude increase of Q. The slope parameter, n adequately describes the shear sensitivity of the

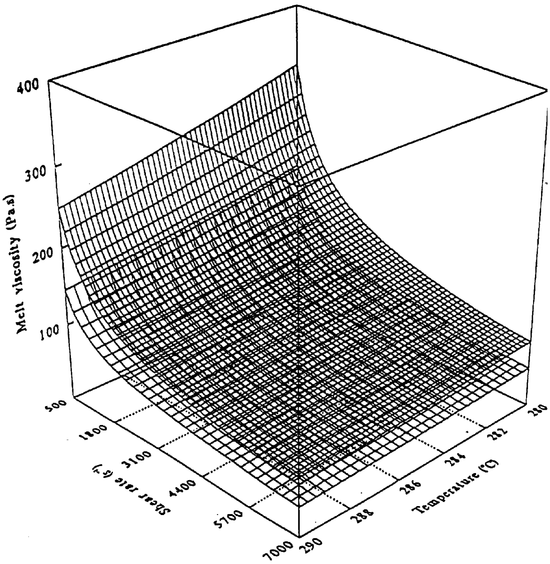


FIGURE 5 Effects of variation of parameter A on melt viscosity. Upper surface: $A = 12 \times 10^6$, lower surface: $A = 7 \times 10^6$, $E = -0.22$, and $n = 0.5$ for both surfaces.

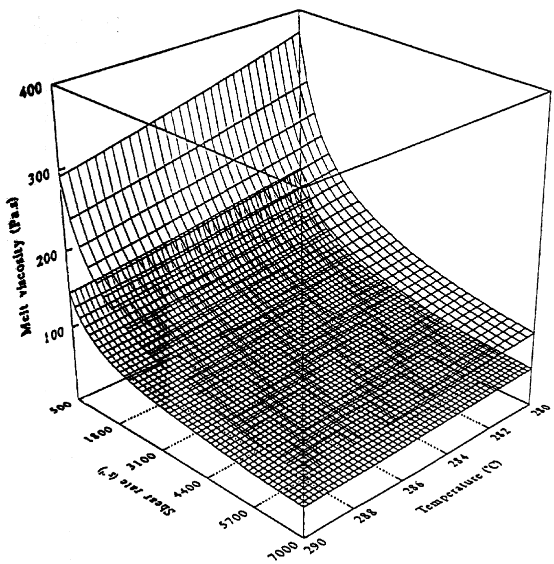


FIGURE 6 Effects of variation of parameter E on melt viscosity. Upper surface: $E = -0.20$, lower surface: $E = 0.22$, $A = 7 \times 10^6$, and $n = 0.5$ for both surfaces.

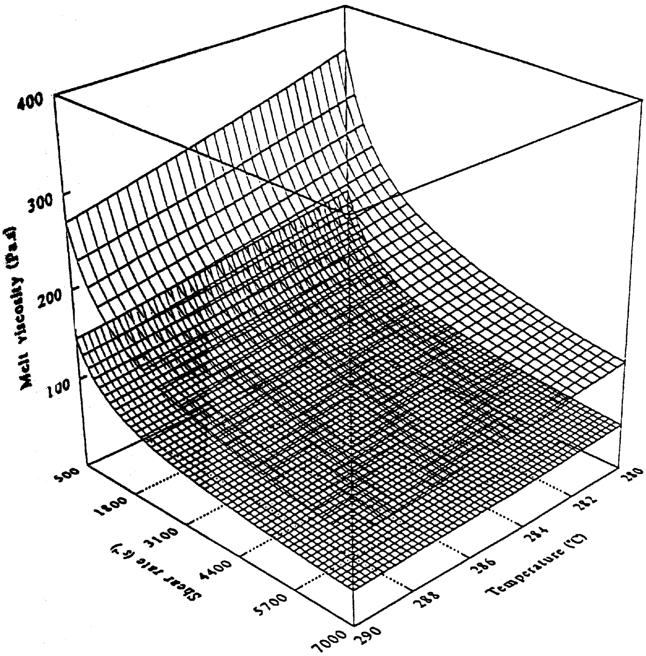


FIGURE 7 Effects of variation of parameter n on melt viscosity. Upper surface: n = 0.60, lower surface: n = 0.50, $A = 7 \times 10^6$, and E = 0.22 for both surfaces.

response surface. The significance of n is therefore illustrated in Figure 7.

CONCLUSIONS

The melt viscosity behaviour of a number of commercial polyamide compounds has been successfully modelled over shear rate and temperature ranges characteristic of injection moulding processes. The estimated parameters are all significant and can be used to determine the nature and extent of the polyamide melt viscosity variations which result from changes in polyamide molecular mass, chemical structure and the addition of different types and levels of additives and fillers.

It was established that for the polyamide compounds investigated, melt viscosity is a function of shear rate at constant temperature in the shear rate range between 500–7000 s^{-1} and this function is appropriately described by Eq. (4), in this shear rate range region. On the other hand, melt viscosity as a function of temperature at constant shear rate is adequately described by Eq. (5), while Eq. (6) effectively describes, the response surface melt viscosity as a function of both the shear rate and temperature. The parameters A, E and n are highly inter-related, as they all affect the average melt viscosity, temperature sensitivity and shear sensitivity, to some degree.

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Appedix: Original polyamide resins data with some melt viscosity properties from Du Pont database[9]

Commercial name	Resin grade no	Shear rate (S^{-1})	Lower temp. (°C)	Middle temp. (°C)	Upper temp. (°C)	Higher melt visc. (mPa.s)	Middle melt visc. (mPa.s)	Lower melt visc. (mPa.s)	Nylon type	%& filler type level
Z73G30BK261	01	500	250	270	290	659	373	212	PA6	30 gr
Z73G30BK261	01	700	250	270	290	550	386	183	PA6	30 gr
Z73G30BK261	01	1000	250	270	290	452	264	156	PA6	30 gr
Z73G30BK261	01	2000	250	270	290	303	181	110	PA6	30 gr
Z73G30BK261	01	5000	250	270	290	175	107	66	PA6	30 gr
Z73G30BK261	01	7000	250	270	290	142	87	55	PA6	30 gr
Z73G30THSL	02	500	250	270	290	414	285	200	PA6	30 gr/im/hs
Z73G30THSL	02	700	250	270	290	352	245	173	PA6	30 gr/im/hs
Z73G30THSL	02	1000	250	270	290	296	207	148	PA6	30 gr/im/hs
Z73G30THSL	02	2000	250	270	290	207	147	106	PA6	30 gr/im/hs
Z73G30THSL	02	5000	250	270	290	126	90	66	PA6	30 gr/im/hs
Z73G30THSL	02	7000	250	270	290	104	75	55	PA6	30 gr/im/hs
Z73G45NC10	03	500	250	270	290	496	316	213	PA6	45 gr
Z73G45NC10	03	700	250	270	290	395	270	184	PA6	45 gr
Z73G45NC10	03	1000	250	270	290	328	226	157	PA6	45 gr
Z73G45NC10	03	2000	250	270	290	226	159	112	PA6	45 gr
Z73G45NC10	03	5000	250	270	290	136	97	70	PA6	45 gr
Z73G45NC10	03	7000	250	270	290	113	81	59	PA6	45 gr
Z73G50BK264	04	500	250	270	290	543	336	209	PA6	50 gr/hs
Z73G50BK264	04	700	250	270	290	458	288	182	PA6	50 gr/hs
Z73G50BK264	04	1000	250	270	290	381	242	156	PA6	50 gr/hs
Z73G50BK264	04	2000	250	270	290	262	170	112	PA6	50 gr/hs
Z73G50BK264	04	5000	250	270	290	155	103	69	PA6	50 gr/hs
Z73G50BK264	04	7000	250	270	290	128	85	58	PA6	50 gr/hs
M13T2GY282	05	500	250	270	290	375	240	158	PA66	30 mr/im

(Continued)

Appendix (Continued)

Commercial name	Resin grade no	Shear rate (S^{-1})	Lower temp. (°C)	Middle temp. (°C)	Upper temp. (°C)	Higher melt visc. (mPa.s)	Middle melt visc. (mPa.s)	Lower melt visc. (mPa.s)	Nylon type	%& filler type level
M13T2GY282	05	700	250	270	290	333	213	141	PA66	30 mr/im
M13T2GY282	05	1000	250	270	290	290	186	123	PA66	30 mr/im
M13T2GY282	05	2000	250	270	290	215	138	92	PA66	30 mr/im
M13T2GY282	05	5000	250	270	290	138	89	59	PA66	30 mr/im
M13T2GY282	05	7000	250	270	290	116	75	50	PA66	30 mr/im
M10B140NC10	06	500	275	295	315	217	112	61	PA66	40 mr
M10B140NC10	06	700	275	295	315	201	104	57	PA66	40 mr
M10B140NC10	06	1000	275	295	315	182	95	52	PA66	40 mr
M10B140NC10	06	2000	275	295	315	144	76	42	PA66	40 mr
M10B140NC10	06	5000	275	295	315	96	52	29	PA66	40 mr
M10B140NC10	06	7000	275	295	315	81	44	25	PA66	40 mr
M11C140NC10	07	500	275	295	315	490	244	119	PA66/6	40 mr/im
M11C140NC10	07	700	275	295	315	434	225	113	PA66/6	40 mr/im
M11C140NC10	07	1000	275	295	315	374	202	105	PA66/6	40 mr/im
M11C140NC10	07	2000	275	295	315	264	156	87	PA66/6	40 mr/im
M11C140NC10	07	5000	275	295	315	149	97	61	PA66/6	40 mr/im
M11C140NC10	07	7000	275	295	315	118	79	51	PA66/6	40 mr/im
MEFE6052NC10	08	500	275	295	315	482	217	104	PA66	50 mica
MEFE6052NC10	08	700	275	295	315	423	191	91	PA66	50 mica
MEFE6052NC10	08	1000	275	295	315	365	166	79	PA66	50 mica
MEFE6052NC10	08	2000	275	295	315	268	122	59	PA66	50 mica
MEFE6052NC10	08	5000	275	295	315	171	78	38	PA66	50 mica
MEFE6052NC10	08	7000	275	295	315	144	66	32	PA66	50 mica
MEFE6053BK16	09	500	275	295	315	383	189	94	PA66	40 mr/gr
MEFE6053BK16	09	700	275	295	315	334	189	86	PA66	40 mr/gr
MEFE6053BK16	09	1000	275	295	315	286	148	77	PA66	40 mr/gr

MEFE6053BK16	09	2000	275	295	315	205	205	60	PA66	40 mr/gr
MEFE6053BK16	09	5000	275	295	315	127	127	40	PA66	40 mr/gr
MEFE6053BK16	09	7000	275	295	315	59	59	34	PA66	40 mr/gr
ZE103HSLCNC1	10	500	275	295	315	110	77	54	PA66	uf
ZE103HSLCNC1	10	700	275	295	315	104	74	52	PA66	uf
ZE103HSLCNC1	10	1000	275	295	315	95	70	50	PA66	uf
ZE103HSLCNC1	10	2000	275	295	315	77	59	44	PA66	uf
ZE103HSLCNC1	10	5000	275	295	315	55	43	34	PA66	uf
ZE103HSLCNC1	10	7000	275	295	315	48	38	30	PA66	uf
0101LNC10	11	500	275	295	315	110	77	54	PA66	uf
0101LNC10	11	700	275	295	315	104	74	52	PA66	uf
0101LNC10	11	1000	275	295	315	95	70	50	PA66	uf
0101LNC10	11	2000	275	295	315	77	59	44	PA66	uf
0101LNC10	11	5000	275	295	315	55	43	34	PA66	uf
0101LNC10	11	7000	275	295	315	48	38	30	PA66	uf
0101FNC10	12	500	275	295	315	110	77	54	PA66	uf
0101FNC10	12	700	275	295	315	104	74	52	PA66	uf
0101FNC10	12	1000	275	295	315	95	70	50	PA66	uf
0101FNC10	12	2000	275	295	315	77	59	44	PA66	uf
0101FNC10	12	5000	275	295	315	55	43	34	PA66	uf
0101FNC10	12	7000	275	295	315	48	38	30	PA66	uf
ZEFE8018NC10	13	500	275	295	315	299	192	126	PA66	uf/im/uvs
ZEFE8018NC10	13	700	275	295	315	260	169	112	PA66	uf/im/uvs
ZEFE8018NC10	13	1000	275	295	315	146	146	98	PA66	uf/im/uvs
ZEFE8018NC10	13	2000	275	295	315	108	108	73	PA66	uf/im/uvs
ZEFE8018NC10	13	5000	275	295	315	70	70	48	PA66	uf/im/uvs
ZEFE8018NC10	13	7000	275	295	315	59	59	41	PA66	uf/im/uvs
Z80G33HS1LNC	14	500	275	290	310	575	391	240	PA66	33 gr/im
Z80G33HS1LNC	14	700	275	290	310	482	327	201	PA66	33 gr/im
Z80G33HS1LNC	14	1000	275	290	310	399	271	166	PA66	33 gr/im
Z80G33HS1LNC	14	2000	275	290	310	275	187	114	PA66	33 gr/im

(Continued)

Appendix (Continued)

Commercial name	Resin grade no	Shear rate (S^{-1})	Lower temp. (°C)	Middle temp. (°C)	Upper temp. (°C)	Higher melt visc. (mPa.s)	Middle melt visc. (mPa.s)	Lower melt visc. (mPa.s)	Nylon type	%& filler type level
Z80G33HS1LNC	14	5000	275	290	310	168	114	69	PA66	33 gr/im
Z80G33HS1LNC	14	7000	275	290	310	140	94	58	PA66	33 gr/im
Z73G30NC10	15	500	250	270	290	396	278	199	PA6	30 gr
Z73G30NC10	15	700	250	270	290	338	240	173	PA6	30 gr
Z73G30NC10	15	1000	250	270	290	285	203	147	PA6	30 gr
Z73G30NC10	15	2000	250	270	290	200	144	106	PA6	30 gr
Z73G30NC10	15	5000	250	270	290	123	88	65	PA6	30 gr
Z73G30NC10	15	7000	250	270	290	101	73	55	PA6	30 gr
Z70G20HSLNC1	16	500	275	290	305	262	165	106	PA66	20 gr
Z70G20HSLNC1	16	700	275	290	305	229	145	93	PA66	20 gr
Z70G20HSLNC1	16	1000	275	290	305	198	126	81	PA66	20 gr
Z70G20HSLNC1	16	2000	275	290	305	149	95	62	PA66	20 gr
Z70G20HSLNC1	16	5000	275	290	305	101	65	42	PA66	20 gr
Z70G20HSLNC1	16	7000	275	290	305	88	56	37	PA66	20 gr
Z70G30HSLNC1	17	500	275	290	305	224	150	102	PA66	30 gr
Z70G30HSLNC1	17	700	275	290	305	198	134	91	PA66	30 gr
Z70G30HSLNC1	17	1000	275	290	305	172	117	81	PA66	30 gr
Z70G30HSLNC1	17	2000	275	290	305	129	89	62	PA66	30 gr
Z70G30HSLNC1	17	5000	275	290	305	86	60	43	PA66	30 gr
Z70G30HSLNC1	17	7000	275	290	305	74	52	37	PA66	30 gr
Z70G35HSLBK3	18	500	275	290	305	275	169	106	PA66	35 gr
Z70G35HSLBK3	18	700	275	290	305	236	146	92	PA66	35 gr
Z70G35HSLBK3	18	1000	275	290	305	201	124	78	PA66	35 gr
Z70G35HSLBK3	18	2000	275	290	305	146	91	58	PA66	35 gr
Z70G35HSLBK3	18	5000	275	290	305	95	59	38	PA66	35 gr
Z70G35HSLBK3	18	7000	275	290	305	81	51	32	PA66	35 gr

Z70G35HSLA4B	19	500	275	290	305	187	112	69	PA66	35 gr/im
Z70G35HSLA4B	19	700	275	290	305	160	95	58	PA66	35 gr/im
Z70G35HSLA4B	19	1000	275	290	305	135	80	49	PA66	35 gr/im
Z70G35HSLA4B	19	2000	275	290	305	97	57	35	PA66	35 gr/im
Z70G35HSLA4B	19	5000	275	290	305	62	37	22	PA66	35 gr/im
Z70G35HSLA4B	19	7000	275	290	305	53	31	19	PA66	35 gr/im
Z70G43LNC10>	20	500	275	290	305	292	177	110	PA66	43 gr
Z70G43LNC10>	20	700	275	290	305	252	153	95	PA66	43 gr
Z70G43LNC10>	20	1000	275	290	305	216	131	81	PA66	43 gr
Z70G43LNC10>	20	2000	275	290	305	159	96	60	PA66	43 gr
Z70G43LNC10>	20	5000	275	290	305	106	64	40	PA66	43 gr
Z70G43LNC10>	20	7000	275	290	305	91	55	34	PA66	43 gr
Z79G13HSLBK3	21	500	275	290	305	238	173	128	PA66	13 gr/im
Z79G13HSLBK3	21	700	275	290	305	207	151	112	PA66	13 gr/im
Z79G13HSLBK3	21	1000	275	290	305	177	130	97	PA66	13 gr/im
Z79G13HSLBK3	21	2000	275	290	305	129	95	71	PA66	13 gr/im
Z79G13HSLBK3	21	5000	275	290	305	83	61	46	PA66	13 gr/im
Z79G13HSLBK3	21	7000	275	290	305	70	52	39	PA66	13 gr/im
0135FNC10	22	500	275	290	305	124	76	47	PA66	uf/nucl
0135FNC10	22	700	275	290	305	117	73	46	PA66	uf/nucl
0135FNC10	22	1000	275	290	305	109	70	44	PA66	uf/nucl
0135FNC10	22	2000	275	290	305	90	60	40	PA66	uf/nucl
0135FNC10	22	5000	275	290	305	62	45	32	PA66	uf/nucl
0135FNC10	22	7000	275	290	305	53	39	28	PA66	uf/nucl
Z408NC10	23	500	275	290	305	143	96	64	PA66	uf/im
Z408NC10	23	700	275	290	305	130	89	60	PA66	uf/im
Z408NC10	23	1000	275	290	305	116	81	56	PA66	uf/im
Z408NC10	23	2000	275	290	305	89	65	46	PA66	uf/im
Z408NC10	23	5000	275	290	305	60	45	34	PA66	uf/im
Z408NC10	23	7000	275	290	305	51	39	29	PA66	uf/im
ZE42NC10	24	500	275	290	305	806	593	504	PA66	uf/hv

(Continued)

Appendix (Continued)

Commercial name	Resin grade no	Shear rate (S^{-1})	Lower temp. (°C)	Middle temp. (°C)	Upper temp. (°C)	Higher melt visc. (mPa.s)	Middle melt visc. (mPa.s)	Lower melt visc. (mPa.s)	Nylon type	%& filler type level
ZE42NC10	24	700	275	290	305	664	501	431	PA66	uf/hv
ZE42NC10	24	1000	275	290	305	535	413	360	PA66	uf/hv
ZE42NC10	24	2000	275	290	305	342	274	243	PA66	uf/hv
ZE42NC10	24	5000	275	290	305	182	150	136	PA66	uf/hv
ZE42NC10	24	7000	275	290	305	144	119	109	PA66	uf/hv
Z490NC10	26	500	275	290	305	251	162	104	PA66	uf
Z490NC10	26	700	275	290	305	221	146	95	PA66	uf
Z490NC10	26	1000	275	290	305	192	129	86	PA66	uf
Z490NC10	26	2000	275	290	305	142	98	67	PA66	uf
Z490NC10	26	5000	275	290	305	92	65	46	PA66	uf
Z490NC10	26	7000	275	290	305	77	55	40	PA66	uf
ZST801LNC	27	500	275	290	305	330	223	151	PA66	uf/im
ZST801LNC	27	700	275	290	305	284	193	132	PA66	uf/im
ZST801LNC	27	1000	275	290	305	241	166	114	PA66	uf/im
ZST801LNC	27	2000	275	290	305	175	121	85	PA66	uf/im
ZST801LNC	27	5000	275	290	305	113	79	56	PA66	uf/im
ZST801LNC	27	7000	275	290	305	96	68	48	PA66	uf/im

Abbreviations:
gr = glass-reinforced, hv = light stabilized.
im = impact-modified, uf = unfilled polyamide resin.
mr = mineral-reinforced, nucl. = nucleated polyamide.
mica = mica-reinforced, PA6 = polyamide 6.
hs = heat stabilized, PA66 = polyamide 66.
uvs = ultra violet stabilized, PA66/6 = polyamide copolymer.