MOLECULAR WEIGHT DISTRIBUTION OF STYRENE POLYMERIZATION IN A STARVED FEED REACTOR

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ABSTRACT

A starved feed reactor is a semi-batch polymerization reactor where initiator and monomer are fed slowly into a fixed amount of solvent. The polymerization is carried out isothermally at elevated temperatures. Initiator decomposes instantaneously and monomer polymerizes immediately. The molecular weight and molecular weight distribution are effectively controlled by the feed ratio of monomer to initiator. This paper presents a study on the molecular weight distribution of styrene polymerization in a starved feed reactor. The molecular weight distribution model parameters are regressed with the help of experimental data. Although the solids fraction in the starved feed reactor is high (>50%), the viscosity is not high, and the "gel effect" is weak because of the lower molecular weight of the products. We found that the termination rate constant is a power function of the molecular weight, the radicals terminate via 100% combination, and the thermal initiation can be neglected although the reaction temperature is high. The calculated results indicate that in the starved feed reactor, the long-chain assumption needs modification so that a more accurate model can be set up.

KEYWORDS

starved feed reactor, molecular weight distribution, styrene, radical polymerization

INTRODUCTION

A starved feed reactor is a semi-batch polymerization reactor in which the initiator and the monomer are fed continuously. The molecular weights of the production in the reactor can be effectively controlled by the ratio of the flow rates of the monomer and the initiator. The low molecular weight polymer can be used in the high-solids fraction coatings directly. In this kind of reactor, the flow rates of the reactants are very low, and both the amount of initiator used and the reaction temperature are higher than usual, so the initiator fed into the reactor decomposes instantaneously, and the monomer reacts instantaneously. In an ideal situation, the concentrations of the initiator and the monomer in the reactor are very low, maybe zero. As the reactor is thus in a "starved" status for reactants, the molecular weight is very low (usually the degree of the polymerization 30~70).

The molecular weight distribution (MWD) of stryrene polymerization in a starved feed reactor is not the same as that in a normal reactor. Much literature has been published on the polymerization kinetics and MWD, but reports on the kinetics and MWD in a starved feed reactor have not yet emerged. This paper presents a study on the kinetics and MWD in a starved feed reactor.

MWD model in a starved feed reactor

In a radical polymerization, an initiator decomposes to form primary radicals:

$$I \xrightarrow{k_d} 2R_0 \bullet$$

Meanwhile, the monomer may also form a primary radical at high temperature:

Thermal initiation:
$$3M \xrightarrow{\bar{k_i}} R_0$$
.

The primary radical R_0 • reacts with the monomer to form the monomer radical:

$$R_0 \cdot + M \rightarrow R_1 \cdot$$

The radical species then attacks the monomer to form a growing macroradical chain:

$$R_1 \circ + M \xrightarrow{k_p} R_2 \circ$$

$$R_i \circ + M \xrightarrow{k_p} R_{i+1} \circ$$

Two propagating radicals can terminate, forming a polymer:

$$R_n \cdot + R_m \cdot \xrightarrow{k_1} Polymer$$

For a typical polymerization in this reactor, the relative contribution of the chain transfer reaction to the initiator, solvent, monomer, and polymer is negligible. Thermal initiation may exist, however, at high temperature, so the initiation rate may be written in the following form:

$$R_{I} = R_{IJ} + R_{IT} = 2 \int k_{d} [I] + 2 \overline{k}_{i} [M]^{3}$$

where \vec{k}_i is the thermal initiating rate constant, the value can be found in reference /1/:

$$\bar{k}_i = 2.19 \times 10^5 \exp(-13810/T)$$
, T/K

 K_d is the decomposing rate constant of the initiator, for AIBN:

$$K_d = 1.6248 \times 10^5 \exp(-1.551 \times 10^4 / RT)$$
, T/K

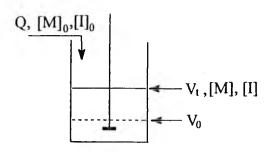


Fig. 1: Flow chart of a starved feed reactor.

Figure 1 is a flow chart of a starved feed reactor, the initial solvent charged to the reactor is V_0 , the initiator solution and the monomer are fed into the reactor at a constant flow rate of Q, and their respective concentrations in the feeding stream are $[I]_0$ and $[M]_0$. The material balance for the initiator is

$$\frac{d\{Q[I]\}}{dt} = [I]_0 Q - k_a [I] V_t \tag{1}$$

Integration of Eq. (1) yields

$$[I] = \frac{Q[I]_0}{Vt} \cdot \frac{1}{k_d} \cdot \{1 - \exp(-k_d t)\}$$
 (2)

The material balance for radicals is

$$\frac{d[R_1 \bullet]}{dt} = R_1 - k_p [M] [R_1 \bullet] - k_i [R_1 \bullet] \left(\frac{R_I}{k_I}\right)^{\frac{1}{2}} = 0$$
 (3a)

$$\frac{d[R_i \bullet]}{dt} = k_p[M][R_{i-1} \bullet] - k_p[M][R_i \bullet] - k_t[R_i \bullet] \left(\frac{R_I}{k_t}\right)^{\frac{1}{2}} = 0$$
 (3b)

Noting that

$$J = \frac{\left(k_p/\sqrt{k_t}\right)[M]}{\left(k_p/\sqrt{k_t}\right)[M] + \sqrt{R_t}} \tag{4}$$

Rewriting Eqs. (3a) and (3b), the following can be obtained.

$$R_1 \cdot = \left(\frac{R_I}{k_I}\right)^{\frac{1}{2}} \left(1 - J\right) \tag{5a}$$

$$R_{i} = \left(\frac{R_{I}}{k_{L}}\right)^{\frac{1}{2}} (1 - J)^{i-1}$$
 (5b)

For styrene polymerization at low temperature (<100°C), the combination termination is 100% /2/, but reportedly a part of disproportionation termination occurs at an elevated temperature (>100°C) /3/, so the material balance for a polymer in a starved feed reactor is

$$\frac{d[P_r]}{dt} = \frac{1}{2} k_{tc} \sum_{j=1}^{r-1} [R_{r-j} \bullet] [R_j \bullet] + k_{td} [R_r \bullet] \sum_{j=1}^{\infty} [R_j \bullet] \qquad r \ge 2$$
 (6)

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Combining Eqs. (5a), (5b), and (6) yields

$$\frac{d[P_r]}{dt} = R_1 J^{r-2} (1 - J) \left\{ \frac{1}{2} (1 - \phi)(r - 1)(1 - J) + \phi J^2 \right\} \qquad r \ge 2$$
 (7)

where

$$\phi = \frac{k_{td}}{k_{tc} + k_{td}} \quad .$$

Equation (7) is the molecular weight distribution model for styrene polymerization is a starved feed reactor.

The monomer balance in the system is

$$\frac{d\{V_t[M]\}}{dt} = Q[M]_0 - R_{POL}V_t \tag{8}$$

where R_{POL} represents the rate of monomer consumption through a reaction with primary radicals (in the initiation step) and macro radicals (in the propagation step), R_{POL} can be written as follows /4/:

$$R_{POL} = R_I + R_P \tag{9}$$

In Eq. (9), R_P represents the propagation rate,

$$R_P = k_p[M] \sum_{i=1}^{\infty} \left[RM_i \bullet \right] = k_p[M] \left(\frac{R_I}{k_t} \right)^{\frac{1}{2}}$$
 (10)

Substituting Eq. (10) into Eq. (9), we can obtain

$$R_{POL} = \sqrt{R_I} \left\{ \sqrt{R_I} + \left(k_p / \sqrt{k_1} \right) [M] \right\}$$
 (11)

Substituting Eq. (4) into Eq. (11), we can obtain

$$R_{POL} = \frac{1}{1 - J} \cdot R_I \tag{12}$$

So, the material balance for the monomer is

$$\frac{d\{V_t[M]\}}{dt} = Q[M]_0 - \frac{1}{1 - J} \cdot R_I \cdot V_t \tag{13a}$$

Equation (13) can be rewritten as follows:

$$\frac{dn_m}{dt} = \dot{n}_m - \frac{1}{1 - J} \cdot R_I \cdot V_t \tag{13b}$$

The monomer concentration in a starved feed reactor can be calculated from Eq. (13a) or (13b). The MWD model contains the following parameters:

- the initiator efficiency f,
- the disproportion termination fraction ϕ ,
- and the lumped rate constant $k_p / \sqrt{k_t}$.

In a wide range of viscosity, the propagation rate constant, k_p , keeps constant /5/, but the termination rate constant, k_t , has a relation to the viscosity in the system and to the polymer chain length. This effect was first reportedby O'Dricoll *et al.* /6/ and has since been confirmed and modeled by other researchers /7-10/. Models of this type take the form (assuming no depropagation):

$$\frac{k_t}{k_t^0} = (x_n)^{-\alpha} \tag{14}$$

This chain length dependence reflects the fact that the termination step in the initial stages of the reaction is controlled by the rate of segmental diffusion of the macroradical chain ends. It has been suggested that the chain length dependence of the termination rate constant can be correlated to the cumulative average degree of polymerization, such that

$$\frac{k_t}{k_t^0} = \left(\overline{X}_n\right)^{-2\alpha} \tag{15}$$

The argument supporting this conclusion is that macro-radicals undergoing segmental diffusion of chain ends are affected by the presence of the surrounding polymer, which reduces the rate of termination in the system.

EXPERIMENTAL AND ANALYSES

Purification of reactants

The respective monomer (styrene, STY) and solvent (2-heptanone, also called methyl amyl ketone of MAK) were washed with 10% aqueous NaOH to remove inhibitors, antioxidants, and impurities. The organic phase was

washed with deionized water until it reached neutrality (pH=7.0) and then dried overnight over calcium anhydride. Then they were distilled at reduced pressure at a reflux temperature of $<25^{\circ}$ C. The head and tail fractions were excluded, and the middle fractions were stored in scaled flasks under refrigeration (-10° C) and used within 24 h.

The initiator (2'2'-azobisisobutyronitrile, AIBN, was used in this work) was double recrystallized from ethanol, using standard procedures, dried under high vacuum, and refrigerated (-10°C) until use.

Procedures

The initiator solution (0.3 mol·L⁻¹) was prepared and kept at room temperature (23°C~25°C). The monomer was stored in a bath of ice water. The initiator and monomer were respectively pumped into the reactor, into which an aliquot (0.2 L) of MAK (the mass was measured and recorded) was charged initially. The reaction temperatures were 100°C, 110°C, 120°C, 130°C, 150°C, and 170°C. Over the course of the run, samples were withdrawn at intervals and placed in vials containing a measured quantity of inhibitor to terminate the reaction. The samples were then analyzed for mass ratio of monomer to solvent (MR), solids fraction, and molecular weight (MW) and MWD.

Sample analyses

The MR was determined by 1002 gas chromatography (GC). The mass fractions of solids (sf) in the samples were determined by gravimetry, and the MW and MWD were determined using a Waters 150C ALC/GPC.

Analysis of experimental data

Figures 2-5 present the experimental results of mass ratio, solids fraction, and MWD versus reaction time at one of the reaction temperatures, in which the reaction time is dimensionless time and is defined as:

$$\tau = t/\theta, \qquad \theta = V_0/Q \tag{16}$$

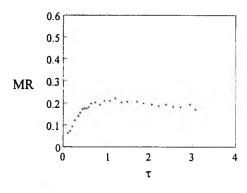


Fig. 2: Mass ratio of STY to MAK vs. reaction time $(T=120^{\circ}\text{C}, \theta = 6554.5\text{s})$

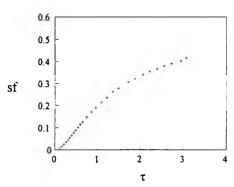


Fig. 3: Solids fraction vs. reaction time (T=120°C, θ =6554.5s)

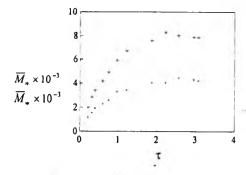


Fig. 4: Average MW vs. reaction time (T =120°C, θ =6554.5s)

■: number average MW

+: weight average MW

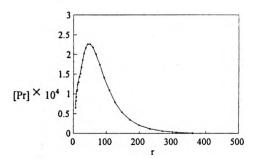


Fig. 5: Polymer concentration vs. chain length (T=120°C, θ = 6554.5s, τ = 2.388)

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 $[P_r] \sim r$, calculated from the data of the initial solvent in the reactor, solvent and monomer feeding rates, solids fraction, and GPC results, is as follows:

$$r = \frac{M_r}{MW_{mon}}$$

$$[P_r] = \frac{(Area)_r}{\sum_{r} (Area)_r} \cdot \frac{m_p}{V_t} \cdot \frac{1}{M_r}$$
(17)

As the volume of the reaction mixture is expected to change with temperature, and the volume decreases with monomers polymerizing to polymers, the volume in the system at time t can be more accurately calculated, such that:

$$V_t = m_s / \rho_s + m_m / \rho_m + m_p / \rho_p,$$

the densities of MAK, STY, and polystyrene (PSTY) at any temperature, are as follows:

$$\begin{split} & \rho_s = \text{A - BT - C/(D - T)} & g \cdot cm^{-3} \\ & \text{A = 1.0823, B = 0.68128 \times 10^{-3}, C = 28.077, D = 711.81,} & \text{T/}^{\circ}\text{K} \\ & \rho_m = 924.0 - 0.918(\text{T - 273.15}) & g \cdot L^{-3} \\ & \rho_p = 1080.8 - 0.605(\text{T - 273.15}) & g \cdot L^{-3} \end{split}$$

The mass of a sample (commonly 0.5-1.5 g) is very much less than that of the contents of the reactor at any time, but the feeding rates of the reactants are small (commonly 1.5 g·min⁻¹). If samples are taken every 5 minutes over the course of a run, then the mass of material fed into the reactor is 7.5 g during the 5 minutes, and the mass ratio of a sample to the material fed into the reactor during the 5 minutes is 6.6%-20%. The mass ration is large, so corrections are made to count for the mass of the samples removed from the reactor; the mass of the solvent, monomer, and polymer in the system at time t can be calculated using the following equations:

$$(m_s)_i = (m_s^0 + \dot{m}_s t_i) - \sum_{j=1}^{i-1} \{sol\}_j$$

$$(m_m)_i = MR_i (m_s)_i$$

$$(m_p)_i = sf_i \cdot \left\{ \left[m_s^0 + (\dot{m}_s + \dot{m}_m)t_i \right] - \sum_{j=1}^{i-1} (\{sol\}_j + \{mon\}_j + \{pol\}_j) \right\}$$
(18)

Regression of model parameters

In the regression of model parameters, the objective function used in this paper is as follows:

$$s = \ln \sum_{i=1}^{N_{MR}} (MR_{model} - MR_{measured})_{i}^{2} + \ln \sum_{i=1}^{N_{ef}} (sf_{model} - sf_{measured})_{i}^{2} + \ln \sum_{i=1}^{N_{\overline{M}_{p}}} (\overline{M}_{nmodel} - \overline{M}_{nmeasured})_{i}^{2} + \ln \sum_{i=1}^{N_{\overline{M}_{p}}} (\overline{M}_{wmodel} - \overline{M}_{wmeasured})_{i}^{2}$$

$$(19)$$

where $MR_{measured}$, $sf_{measured}$, $\overline{M}_{nmeasured}$, $\overline{M}_{wmeasured}$ are the experimental results, and MR_{model} , sf_{model} , \overline{M}_{nmodel} , \overline{M}_{wmodel} are the model values calculated from Eqs. (7) and (13). They are as follows:

$$MR_{model,i} = \frac{m_{m,modeli}}{m_s^0 + \dot{m}_s t_i} , \quad i = 1$$

$$MR_{model,i} = \frac{m_{m,modeli} - \sum_{j=1}^{i-1} \{mon\}_{j}}{m_{s}^{0} + \dot{m}_{s}t_{i} - \sum_{j=1}^{i-1} \{sol\}_{j}} , \quad i \geq 2$$

$$s\bar{f}_{model,i} = \frac{\dot{m}_m t_i - m_{m,modeli}}{m_s^0 + \dot{m}_s t_i + \dot{m}_m t_i} , \quad i = 1$$

$$sf_{model,j} = \frac{\dot{m}_{m}t_{1} - m_{m,modeli} - \sum_{j=1}^{i-1} \{pol\}_{j}}{m_{s}^{0} + \dot{m}_{s}t_{i} + \dot{m}_{m}t_{i} - \sum_{j=1}^{i-1} \left(\{sol\}_{j} + \{mon\}_{j} + \{pol\}_{j} \right)}, \quad i \geq 2$$

$$\overline{M}_{n,modeli} = \frac{\sum_{r=1}^{\infty} ([P_r]_{m,model,j} \cdot r)}{\sum_{r=1}^{\infty} (r)} \cdot MW_{mon}$$

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$$\overline{M}_{w, \int \text{mod } eh} = \frac{\sum_{r=1}^{\infty} \left([P_r]_{m, model, i} \cdot r^2 \right)}{\sum_{r=1}^{\infty} \left([P_r]_{m, model, i} \cdot r \right)} \cdot MW_{mon}$$

where $[P_r]_{model,i}$, $m_{model,i}$ are the results of integration of Eqs. (7) and (13).

DISCUSSION

Model parameters

The model parameters were calculated as shown above, the relation between the lumped rate constant $k_p \sqrt{k_t^0}$ and temperature is as follows:

$$k_p / \sqrt{k_t} = 837.15 \exp\left(-\frac{4060}{T}\right)$$
.

Fig. 6 shows the relationship.

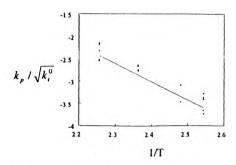


Fig. 6: Rate constant $k_p \sqrt{k_t^0}$ vs. temperature.

In the experimental range, other parameters remain constant, respectively, in which ϕ =0. It can be seen that only combination termination occurs while styrene is polymerizing in a starved feel reactor at an elevated temperature.

Thermal initiation

Using the model parameters, the thermal initiation rate was calculated. The result shows that

$$\frac{R_{I,I}}{R_{I,I} + R_{I,T}} = \frac{2\bar{k}_i[M]^3}{2fk_d[I] + 2\bar{k}_i[M]^3} = 5 \times 10^{-6} \sim 8 \times 10^{-6}$$

In a typical starved feed reactor, the relative amount of initiator used is large, and the monomer concentration is very low, the thermal initiation almost does not occur, although the reaction temperature is high $(100^{\circ}\text{C}\sim170^{\circ}\text{C})$.

Long chain assumption

In setting up a kinetics model of radical polymerization, three assumptions are usually used:

- 1. Identical activate of radicals assumption,
- 2. Steady state assumption,
- 3. Long chain assumption.

The third assumption holds that the degree of polymer is very high, the amount of monomer consumption by primary radicals is lower than that by macro-radicals, so the polymerization rate is equal to the propagation rate. But in a starved feed reactor, the degree of polymer is low, and the relative amount of initiator used is high, so the long chain assumption cannot be used here. The calculated result proves this point. For normal radical polymerization, $R_I/R_p = 10\%\sim50\%$.

CONCLUSION

In a starved feed reactor, the molecular weight can be effectively controlled by the feed rate of the monomer and initiator, the degree of polymer model and a molecular weight distribution model have been set up, and the model parameters were regressed with the help of experimental data.

The relation between the lump rate constant $k_p \sqrt{k_t^0}$ and the temperature can be represented via an Arrhenius equation.

In a typical starved feed reactor, the relative amount of initiator used is large, and the monomer concentration is very low, and thermal initiation almost does not occur. As the amount of monomer consumption by primary radicals is not negligible, long chain assumption cannot be used here.

Although the solids fraction in the starved feed reactor is high, the viscosity is not high, and the "gel effect" is weak because the molecular weight of the products is low. The termination rate constant is a power function of the cumulative average degree of polymerization. In the starved feed reactor, the reaction temperature is high, but the radicals terminate via 100% combination.

GLOSSARY

(Area) _r	differential area in GPC analysis	М	monomer
f	initiator efficiency	\overline{M}_n	number average MW
I	initiator molecule	\overline{M}_{w}	weight average MW
[1]0	initiator concentration in the SFR feedstreams, mol-L	MR	mass ratio monomer to solvent
k _d	interior decomposition rate constant, s ⁻¹	M _r	MW of polymer incorporating "r" monomer units
J	a constant	MW _{mon}	MW of monomer
\overline{k}_i	thermal initiating rate constant $L^2 \cdot mol^{-2} \cdot s^{-1}$	[M] ₀	monomer concentration in the SFR feedstream, mol·L ⁻¹
$\mathbf{k}_{\mathbf{p}}$	propagation rate constant, L·mol ⁻¹ ·s	m _m	mass of monomer in the SFR, g
k _t	termination rate constant, L·mol ⁻¹ ·s ⁻¹	m _p	mass of polymer in the SFR, g
k _{tc}	termination rate constant due to combination, L·mol ⁻¹ s ⁻¹	ms	mass of solvent in the SFR, g
k _{td}	termination rate constant due to disproportionation, L·mol ⁻¹ ·s ⁻¹	m_s^0	mass of initial solvent charged to the SFR, g

GLOSSARY (CONT'D)

\dot{m}_m	mass flow rate of monomer into the SFR, g·s ⁻¹	s	objective function
m _s	mass flow rate of solvent into the SFR, g·s ⁻¹	sf	solids fraction
{mon}	mass of monomer in SFR samples, g	{sol}	mass of solvent in SFR samples,
Ni	no. samples of type i, i represents MR, sf, \overline{M}_n , \overline{M}_w	T	reaction temperature, K
n _m	moles of monomer in the SFR, mol	t	time, s
n _m	molar flow rate of monomer into the SFR, mol s ⁻¹	V ₀	volume of initial solvent charged to the SFR, L
Pr	polymer that has incorporated "r" monomer units	Vt	volume of reaction mixture in the SFR at time t, L
{pol}	mass of polymer in SFR samples, g	Q	volume feed rate of the SFR feed stream, L·s
R	gas constant	Xn	chain length
R_0 •	primary radical	\bar{x}_n	cumulative average degree of polymerization
R ₁ •	radical that has incorporated "i" monomer units	α	a constant
R_1	initiation rate, mol·L ⁻¹ ·s ⁻¹	ф	a fraction of disproportionation
R _{I,I}	initiator initiation rate, mol·L ⁻¹ ·s ⁻¹	θ	replacement time of the SFR, s
R _{I,T}	thermal initiation rate, mol·L ⁻¹ ·s ⁻¹	$\rho_{\rm m}$	density of monomer, g·L ⁻¹
Rp	propagation rate, mol·L ⁻¹ ·s ⁻¹	Рρ	density of polymer, g·L ⁻¹
R _{POL}	rate of monomer consumption in polymerization, $mol \cdot L^{-1} \cdot s^{-1}$	ρs	density of solvent, g·L ⁻¹
r	number of monomer units in polymer chain	τ	dimensionless reaction time

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Thin films and coatings are one of the most pervasive and widespread technologies of the modern world. Applications range from the preservation of metal finishes in industries such as automobiles and watercraft to forming critical dielectric layers in microelectronic structures which are the foundation of the computer and consumer electronics industries. In all of these applications the adhesion of the coating to its substrate is critical to the coating's performance, reliability and durability. Thus the ability to accurately measure the adhesion of coatings to surfaces is a crucial part of the development and manufacturing process of coatings and films. In addition, the ability to make accurate adhesion measurements requires a fundamental understanding of the physics, chemistry and mechanics of thin films and coatings. This symposium is the natural follow on to the first international conference on this topic held in 1992 in Boston. This, the second symposium, will follow up on the latest developments in this tremendously active field. The primary focus of this meeting will be to provide a forum for the discussion of cutting edge advancements in the field and to review and consolidate the accomplishments which have been achieved thus far.

TOPICS OF INTEREST INCLUDE:

- Adhesion measurements in quality control and manufacturing
- Adhesion measurements in support of coating process research and development
- Adhesion measurement instrumentation for laboratory and manufacturing environments

FUNDAMENTAL ASPECTS OF ADHESION MEASUREMENT

- Mechanics of adhesion testing, the role of film stresses
- Fracture mechanics of adhesion testing
- Physico-chemical aspects of adhesion testing, the role of film morphology and chemistry

ADVANCED TEST AND DATA ANALYSIS METHODS

- Qualitative, semiquantitative and fully quantitative analysis methods.
- Novel test methods: laser spallation, internal friction, electromagnetic,... etc.
- Thermodynamic aspects of adhesion testing, (energy flow and balance, calorimetry, ... etc)

This symposium is being organized under the direction of Dr. K. L. Mittal, Editor, Journal of Adhesion Science and Technology by MST Conferences, LLC. A proceedings volume is planned for this symposium and further details will be provided in due course. Please notify the conference chairman of your intentions to present a paper as early as possible. An abstract of about 200 words should be earn by Mey 30, 1999 to the conference chairman by any of the following methods:

E-mail: rhlacombe@compuserve.com

FAX: 212-656-1016

Regular mail:

Dr. Robert H. Lacombe. Conference Chairman. 3 Hammer Drive Hopewell Junction, NY 12533

Contact by phone: 914-227-7026

Full conference details and registration via the Internet will be maintained on our web site:

http://mstconf.com/adhmeas.htm

Or mail response form below to conference chairman at address above.

SECOND INTERNATIONAL SYMPOSIUM ON ADHESION MEASUREMENT OF THIN FILMS AND COATINGS I PLAN TO: ATTEND PRESENT A PAPER TENTATIVE TITLE:				
ADDRESS:	TELEPHONE:			
	FAX:			
	E-MAIL:			

CALL FOR PAPERS INTERNATIONAL SYMPOSIUM ON ADHESION ASPECTS OF THIN FILMS

OCTOBER 28-29, 1999 SHERATON NEWARK AIRPORT NEWARK, NJ

This is a companion symposium to the SECOND INTERNATIONAL SYMPOSIUM ON ADHESION MEASUREMENT OF THIN FILMS AND COATINGS. This symposium will deal with adhesion aspects of all types of thin films. The symposium will focus on a range of adhesion related concerns dealing with durability and reliability. Particular issues include the determination of the locus of adhesion failure, film-substrate interactions, bond durability against moisture and other deleterious factors, solvent swelling effects and the role of residual stresses on film performance and reliability. This symposium will follow up on the latest developments in this tremendously active field. The primary focus of this meeting will be to provide a forum for the discussion of cutting edge advancements in the field and to review and consolidate the accomplishments which have been achieved thus far.

BOTH ORGANIC AND INORGANIC THIN FILMS ARE OF INTEREST IRRESPECTIVE OF DEPOSITION HETHOD

TOPICS OF INTEREST INCLUDE:

- Factors influencing adhesion Residual stress, mechanical properties, contamination ... etc.
- Bond durability, corrosion prevention
- Adhesion promoters

POLYMERIC FILMS

- Plasma polymerized films
- Photoresists
- Organic insulators
- Barrier layers
- Effects of aging and environment on adhesion

GENERAL SYSTEMS

- Polymer to metal and metal to polymer adhesion
- Multilevel laminates involving glass, ceramic, metal and polymer thin films

FUNDAMENTAL ISSUES

- Role of surface chemistry, wettability and morphology
- Fundamental adhesion mechanisms including film/substrate interactions

This symposium is being organized under the direction of Dr. K. L. Mittal, Editor, Journal of Adhesion Science and Technology by MST Conferences, LLC. A proceedings volume is planned for this symposium and further details will be provided in due course. Please notify the conference chairman of your intentions to present a paper as early as possible. An abstract of about 200 words should be sent by May 30, 1999 to the conference chairman by any of the following methods:

E-mail: rhlacombe@compuserve.com

FAX: 212-656-1016

Regular mail:

Dr. Robert H. Lacombe. Conference Chairman. 3 Hammer Drive Hopewell Junction, NY 12533

Contact by phone: 914-227-7026

Full conference details and registration via the Internet will be maintained on our web site:

http://mstconf.com/adhfilm.htm

Or mail response form below to conference chairman at address above.

INTERNATIONAL SYMPOSIUM ON ADHESION ASPECTS OF THIN FILMS I PLAN TO: ATTIEND PRESENT A PAPER TENTATIVE TITLE;					
ADDRESS:	TELEPHONE:				
	FAX:				
	E-MAIL:				

FREUND PUBLISHING HOUSE LTD.

Suite 500, Chesham House, 150 Regent Street, London W1R 5FA, England

TITLES OF JOURNALS AND BOOKS IN THE MATERIALS SCIENCES AND ENGINEERING

JOURNALS

CORROSION REVIEWS

Editor: M. Schorr 1999 subscription: \$340 including air mail (6 issues) ISSN 0334-6005

Corrosion Reviews is an international quarterly periodical devoted to the theoretical and practical aspects of corrosion science, engineering and technology. It provides a forum for developments in fundamental and applied corrosion research and it covers the corrosion field in its broadest sense. Corrosion Reviews publishes research articles, short communications and reviews.

HIGH TEMPERATURE MATERIALS AND PROCESSES

Editors: A. Rosen and Y. Waseda 1999 subscription: \$340 including air mail ISSN 0334-6455

High Temperature Materials and Processes is devoted to presenting a better understanding of process fundamentals at high temperatures and changes occurring in related technology. It covers the rapidly expanding field of high temperature applications and behavior of metals and alloys, intermetallic compounds, etc., as well as oxidation and environmental attack, high temperature corrosion, designs using high temperature materials, processes for achieving high temperatures, and so

INTERNATIONAL JOURNAL OF TURBO AND JET **ENGINES**

Editor B. Gal-Or 1999 subscription: \$320 including air mail ISSN 0334-0082

The aim of the International Journal of Turbo and Jet Engines is to contribute to the advancement of the science and technology of air, land and marine applications of turbo and jet engines. The journal presents original papers and reviews on research, development and applications covering topics like fluid dynamics in turbomachinery, aerothermodynamics, combustion and alternative fuels, materials and modern processing methods, engine control and instrumentation, structures, vibration and noise reduction, computer software and CAD/CAM, engine tests, systems engineering of transmissions, heat exchangers, APUS, fuel systems instrumentation and engine-vehicle interfaces, etc.

JOURNAL OF THE MECHANICAL BEHAVIOR OF MATERIALS

Editors: E.C. Aifantis and S. Murakami 1999 subscription: \$340 including air mail ISSN 0334-8938

The Journal covers all modern engineering materials: metals and alloys, ceramics and glass, polymers and composite materials, wood, elastomers, etc. In addition to regular issues of the Journal, special issues on specific subjects are often published. These contain invited reviews by eminent workers in the field.

JOURNAL OF POLYMER ENGINEERING

Editor: M. Narkis. Co-Editors: R.E. Cohen, A. Siegmann, and J.M. Vergnaud

1999 subscription: \$380 including air mail (6 issues) ISSN 0334-6447

The Journal of Polymer Engineering publishes articles whose main areas of concentration relate to basic research and innovation in polymer processing as well as characteristics and properties of fabricated products including composites and new applications.

REVIEWS IN CHEMICAL ENGINEERING

Editors: D. Luss, N.R. Amundson and A. Marmur 1999 subscription: \$300 including air mail (4 issues) ISSN 0167-9299

The main aim of Reviews in Chemical Engineering is to develop new insights and to promote interest and research activity in chemical engineering and applied chemistry, as well as the application of new developments in these areas. The journal publishes authoritative articles of limited scope by leading chemical engineers, applied scientists mathematicians

SCIENCE AND ENGINEERING OF COMPOSITE MATERIALS

Editor for North America: S.V. Hoa, Editor for Asia: M. Zako, Scientific Editor. LG. Zewl 1999 subscription: \$340 including air mail

Science and Engineering of Composite Materials provides a forum for publication and discussion of all aspects related to the structure and performance under simulated and actual service conditions of composites used for structural applications, in biomedical engineering, in electronics, etc. The publication covers a variety of subjects, like macro- and microstructure of the materials, their mechanics and micromechanics, the interphase, physical and chemical ageing, fatigue, environmental interactions, fracture, etc. The interdisciplinary character of the subject as well as the possible use of composites of all kinds for new and specific applications receives special attention.

ISSN 0334-181X

<u>NEW JOURNAL:</u> INTERNATIONAL JOURNAL FOR MANUFACTURING SCIENCE AND PRODUCTION

Editor-in-chief: J.D. Marinescu, Editor for Europe: ILK. Toenshoff, Editor for Asia and Australia: Ichiro Inasaki 1999 subscription: \$250 including air mail ISSN 0793 6648

The International Journal for Manufacturing Science and Production reports the latest developments and original applications, theoretical research and case studies in all fields of manufacturing engineering. This new quarterly will be of special interest to industrial and manufacturing engineers,

mechanical engineers, production managers, manufacturing managers, strategic planners, government officials with responsibilities for manufacturing, researchers and scientists in manufacturing engineering.

BOOKS

BATCH CRYSTALLIZERS

This book presents an outline of the techniques used in experimental data reduction and analyses of batch crystallizers. A process description based on batch conservation equations describing population, mass and energy balances together with appropriate kinetic events represented by phenomenological models and proper boundary conditions is employed. A number of useful general techniques to extract crystallization kinetics and to assess the crystallizer performance are illustrated with numerical examples.

Author: N.S. Tavare
145 pages, published in 1992
\$80 including air mail
ISBN 965-294-068-2
(Originally published in Paylant In Cl.

(Originally published in Reviews in Chemical Engineering)

BIOLOGICALLY INDUCED CORROSION

This volume contains long, exhaustive reviews by well-known specialists on biologically influenced corrosion problems in water, soil, oil, and the methods employed to detect, locate, identify, control and solve these problems.

The book illustrates the biodeterioration of metals, concrete, plastics, rubber, bitumen and other organic materials by the micro- and macroorganisms present in these environments. The properties of the environment and their interaction with organisms, techniques for determining biocorrosion and current trends in biocorrosion control are also examined.

Editor: M. Schorr
154 pages, published in 1990
570 including air mail
ISBN 965-294-048-8 (Special issue in Corrosion Reviews)

CHEMISTRY AND METALLURGY OF REFRACTORY AND REACTIVE METALS AND MATERIALS: EXTRACTION AND PROCESSING

This two-volume book contains an assortment of authoritative contributions from experts and specialists in the field of retractory and reactive metals. This group of metals played a key role in the development of modern science and technology and particularly in the development of electronic, nuclear and space engineering. The contributions contained in this book are representative of the present situation in the field and are intended to give the reader an appraisal of some recent developments and a general appreciation of the special metallurgical features and aspects of processing of refractory and reactive metals.

Editor: C.K. Gupta
Vol. 1 (259 pages, published in 1991)
Vol. 2 (approx. 330 pages, published end 1992)
\$120 per volume including air mail
ISBN 965-294-053-4
(Special issues in High Temperature Materials and
Processes)

INJECTION-MOLDED FIBER-REINFORCED THER-MOPLASTICS: SELECTED CONTEMPORARY TOPICS
This book presents an overview of recent developments in injection-molded composites with thermoplastic matrices, including fiber orientation prediction by flow simulation, use of fracture mechanics methods at static, dynamic and cyclic loadings as well as estimation and modeling of the fundamental mechanical properties of discontinuous fiber-reinforced

Editor: J. Karger-Kocsts
173 pages, published in 1992
\$80 including air mail
ISBN 965-294-072-0
(Special issue in Journal of Polymer Engineering)

thermoplastic composites.

COMPUTER APPLICATIONS IN CORROSION

The application of artificial intelligence in performing expert functions has provided new paradigms for the integration of knowledge coming from different sources, from simple databases to living human expertise. A new engineering discipline has emerged that specializes in the elicitation and representation of expert knowledge. This book discusses the applications of expert system technology to corrosion prevention.

Editor: P.R. Roberge One volume, published in 1996 \$100.00 including air mail ISSN 0048-7538 (Special issue in Corrosion Reviews)

COMPUTER APPLICATIONS IN CORROSION

The purpose of this book is to show a practical and pragmatic approach in the use of personal computers for solving corrosion problems in chemical plants, power stations and other industrial systems. This volume offers knowledge and experience on database systems, application of expert systems in chemical engineering and corrosion control in industry, and computerized systems for corrosion testing and evaluation and for cathodic protection design and control. The book gives the scientist and engineer an understanding of the current trend in this novel field.

Editors: R.N. Parkins and M. Schorr
184 pages, published in 1987
550 including air mail
ISBN 965-294-050-X (Special issue in Corrosion Reviews)

CORROSION CONTROL IN INDUSTRIAL SYSTEMS

This three-volume book is a collection of basic papers authored by international experts dealing with corrosion control in industrial plants and environments, including their equipment to ensure safety and maximize profit. The emphasis is on the analysis of corrosion failures, their diagnosis and application of preventive and curative remedies to avoid such events in the future.

The book includes practical information on the use of specially developed corrosion-resistant alloys for industrial plants, corrosion control in sugar and alcohol plants; solution of corrosion problems in power plants, corrosion in marine environment, polluted atmospheres and saline waters; corrosion prediction and control in chemical process industries and protection of domestic water systems.

Editor: M. Schorr Three volumes, 672 pages, published in 1990, 1991 and 1993 \$180 including air mail ISBN 965-294-062-3 (Special issue in Corrosion Reviews)

CORROSION CONTROL IN MARINE STRUCTURES AND ENVIRONMENTS

This book presents the reader with a comprehensive collection of papers on the prdiction, prevention, protection and control of corrosion in civil structures, built of reinforced concrete, steels and other metallic and nonmetallic materials in marine environments. This work includes papers on the behaviour of steels, stainless steel and aluminum alloys in seawater service, biocorrosion in fluvial and marine systems, preservation of infrastructure, corrosion in the Cuban caribbean sea, and coating for corresion protection in seawater structures.

Editor: G.H. Duque D. Two volumes, 514 pages, published in 1994 and 1995 \$180 including air mail ISBN 0048-7538 (Special issues in Corrosion Reviews)

CORROSION CONTROL IN POWER STATIONS -THERMAL AND HYDRO-ELECTRIC

This two-volume book provides in-depth, updated information on corrosion control practices in power stations, both thermal and hydroelectric. This work includes most energy generating and utilizing systems: thermal power plants, steam generators, heat exchangers for water cooling in power plants, fossil-fuel industrial boilers, flue gas desulphurization units, electricity transmission and distribution systems, and hydroelectric generating plants. The book deals with methods and techniques for corrosion prevention and protection: painting in hydropower plants, cathodic protection in power line structure foundations, treatment of boiler feed-water and cleaning of steam generators.

Editor: M. Schorr Two volumes, 417 pages, published in 1988 \$100 including air mail ISBN 965-294-049-6 (Special issues in Corrosion Reviews)

CORROSION RESISTANCE OF ALUMINUM ALLOYS

The papers presented in this special issue are a selection of fundamental and applied research works by experts in various aspects of corrosion and its control in the aluminum industry. The reviews include corrosion of aluminum alloys and their control, corrosion of aluminum-based composites, industrial applications of aluminum, research and testing methods, etc. The authors include scientists and engineers from industry and academia.

Editor: Dr. Joseph Zahavi 300 pages, published in 1997 \$110 including air mail ISBN 0048-7358 (Special issue in Corresion Reviews)

CORROSION RESISTANCE OF MAGNESIUM ALLOYS

The papers presented in this special issue are a selection of fundamental and applied research works by experts in various aspects of corrosion and its control in the magnesium industry. The reviews include corrosion of magnesium alloys and their control, corrosion of magnesium-based composites, industrial applications of magnesium alloys, improved surface by laser alloying performance of magnesium anodes in cathodic protection, research and testing methods, etc.

Editor: Prof. Barry Leslie Mordike 226 pages, published in 1998 \$80 including air mail ISBN 0048-7358 (Special issue in Corrosion Reviews)

DESIGN AND MANUFACTURING USING COMPOSITES

An international conference on Design and Manufacturing Using Composites was held in Montreal in August, 1994. Relevant papers from this conference were selected for publication in this special issue.

Editor: Professor Suong V. Hon One volume, 80 pages, published in 1995. \$50,00 including air mail

(Special issue in Science and Engineering of Composite Materials)

GEL ENTRAPMENT AND MICRO-ENCAPSULATION: METHODS, APPLICATIONS AND ENGINEERING PRINCIPLES

The immobilisation of living cells in gel matrices and micro-capsules has evolved into an established technique with numerous applications in fields such as industrial microbiology, pharmaceuticals, food and beverages, biomedical engineering, agriculture and waste treatment. This wide-ranging review fills a gap in the existing literature.

Authors: R.G. Willsert and G.V. Baron One volume, published in 1996. \$120.00 including air mail. (Special issue in Reviews in Chemical Engineering)

GLOBAL FRONTIERS IN MANUFACTURING

The 13th International Conference on Production Research was held in Jerusalem, Israel in 1995. The theme of the Jerusalem conference was "Global Frontiers in Manufacturing". Editors: E.M. Dar-El, R. Karni and Y.T. Herer

One volume, 800 pp., hard cover, published in 1995. \$150.00 including airmail

HIGH ENERGY HEATING SOURCES

As its title implies, this three-volume set deals with many aspects of high energy heating sources. It also deals with many facets of the mechanical behavior of high temperature materials

Editor: L Minkoff Three volumes, 240 pages, published in 1987 \$100 including air mail ISBN 965-294-061-5 (Special issue in High Temperature Materials and Processes)



AND BUSINESS

INTERNATIONAL SYMPOSIUM ON THE SAFETY AND QUALITY OF FOOD AND DRUGS WITH PLASTIC PACKAGINGS

This special issue contains selected lectures from a symposium held in Paris in November, 1993 on the safety and quality of food and drugs with plastic packagings, with special emphasis on the potential migration of additives from packaging into the packed material. Various fields, such as legislation, test experiments, various fields, such as legislation, test experiments, mathematical treatment, and history of the studies are covered mathematical treatment, and history of the studies are covered.

Editor: J.M. Vergnaud

One volume, published in 1995/96

\$110 including air mail

(Special issue in Journal of Polymer Engineering)

LIQUID METALS AND SURFACE TREATMENTS

This book presents 9 review articles on different facets of liquid metal and surface treatments.

Editor, Y. Waseda

71 pages, published in 1989

\$35 including air mail

ISBN 965-294-078-X

(Special issue in High Temperature Materials and

Processes)

MECHANICS AND ACOUSTICS OF GRANULAR MATER-IALS, PART I: STATIC AND QUASI-STATIC STATES

This monograph is the first of two parts on the subject of the mechanics of granular or particulate materials, dealing with granular materials in the static state. It includes discussion of fine powders, of interest in pharmaceuticals.

Author: G. Rosenhouse

One volume, published in 1995

\$60.00 including airmail

ISSN 0167-8299

(Special issue in Reviews in Chemical Engineering)

MULTIPHASE REACTION ENGINEERING FOR FINE CHEMICALS AND PHARMACEUTICALS

This book presents a review of recent experimental and theoretical developments and anticipated needs in the analysis and design of gas-liquid catalytic reactors used in manufacturing processes for fine chemicals and pharmaceuticals.

Authors; P.L. Mills, P.A. Remachandran and R.V. Chandhart

173 pages, published in 1992

\$80 including air mail

ISBN 965-294-067-4

(Originally published in Reviews in Chemical Engineering)

PLASTIC INSTABILITIES IN SOLIDS

The purpose of this book is to present the reader with a survey of some aspects of instabilities in the plastic deformation of solids. Articles by seven international experts in the field highlight the difficulty in combining micro- and mecroscopic aspects of crystal plasticity, the Portevin le Chatelier effect, strain localization induced by either geometrical shearing or chemical reversion of ordered precipitates and instabilities due to the geometrical inhomogeneity of glide,

Editor: G. Saada

145 pages, published in 1989

\$60 including air mail ISBN 965-294-058-5

(Special issue in Journal of the Mechanical Behavior of Materials)

POLYPROPYLENE

This book contains an assortment of articles by specialists in the field of polypropylene

251 pages, published in 1991

\$100 including air mail ISBN 965-294-056-9

(Special issue in Journal of Polymer Engineering)

RARR METALS

This book contains eight authoritative articles on rare metals under high temperature conditions and their processing and applications.

Editor: Y. Waseda

88 pages, published in 1988

\$50 including air mail

ISBN 965-294-060-7 (Special issue in High Temperature Materials and Processes)

RECENT TOPICS ON POLYOLEFINS AND RELATED

This book contains 6 reviews by internationally recognized experts on polyolefins and related blends.

Editor: J. Karger-Kocais

Approx. 120 pages, published 1992.

\$80 including air mail

ISBN 965-294-071-2

(Special issue in Journal of Polymer Engineering)

STRENGTH OF METALS AND ALLOYS

This book contains the proceedings of the Ninth International Conference of Metals and Alloys (ICSMA-9) which was held at the Technion, Israel Institute of Technology in Haifa on July 14-19, 1991. The main theme of the conference was the fundamental physical aspects of strengthening of crystalline materials. Many of the research papers included in this book were contributed by scientists from China, Eastern Europe and the former U.S.S.R.

Editors: D.G. Brandon, R. Chaim and A. Rosen Two volumes, 1174 pages, published in 1991 \$50 including air mail

ISBN 965-294-054-2

PURE METALS PROPERTIES

This book deals with the chemical, physical and mechanical properties of pure metals, e.g. the quantity and character of foreign atoms, fabrication methods, cold-working degree, thermal treatment procedures, grain size, measuring methods, etc., data which differ greatly in various books. Conventional metal handbooks lack data on rare or pure metals, low or high temperatures, etc. These and other important factors are considered in this book. The authors have introduced a new bond parameter (atomic heat capacity) and present the connection of bond parameters with properties of very pure metals.

Editors: A. Buch and V. Leizin To be published in 1999 \$80 including air mail

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