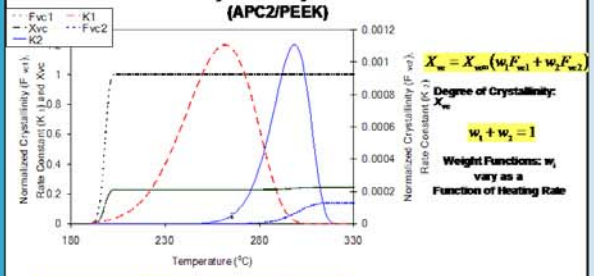
CRYSTALLIZATION KINECTICS BEHAVIOR OF PEEK-BASED COMPOSITES EXPOSED TO HIGH HEATING AND COOLING RATE

J. Tierney and J. W. Gillespie, Jr.

University of Delaware • Center for Composite Materials • Department of Materials Science and Engineering

CRYSTALLIZATION KINETICS GROWTH MODEL

SEFERIS AND VELISARIS (1986)
Dual Mechanisms: Spherulitic and Transcrystalline Crystal Growth (APC2/PEEK)



$$X_w = X_{wm}\left(w_1 F_{w1} + w_2 F_{w2}\right)$$

Degree of Crystallinity: X_w

$$w_1 + w_2 = 1$$

Weight Functions: w_i vary as a Function of Heating Rate

$$K_1(T) = C_{1(i)} T \exp\left\{-\left[\frac{C_{2(i)}}{T - T_g + 51.6} + \frac{C_{3(i)}}{T\left(T_{m(i)} - T\right)^2}\right]\right\}$$

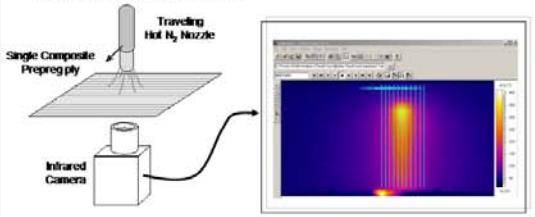
← Rate Constant for each Mechanism: K_i

Normalized Crystallinity: F_i

$$\left[\ln\left(\frac{1}{1-F_{w(i)}}\right)\right]^{1/n} = \left[\ln\left(\frac{1}{1-F_{w(i)_0}}\right)\right]^{1/n} + \int_t^{t+\Delta t}\left[K_i(T(t))\right]^{1/n} dt$$

HOT GAS NOZZLE EXPERIMENT

- Novel experimental technique developed to characterize crystal growth under ultra-high heating and cooling rates.
- Single tow CF/PEEK samples are exposed to high temperature N2 gas using robot.
- Thermal Imaging Camera records surface temperature data.
- Thermal data applied to crystallization kinetics model.
- DSC experiments are carried out on marked specimens to validate model predictions



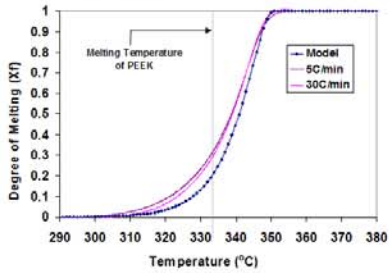
Thermal image of composite prepreg showing data extraction regions

ACKNOWLEDGEMENTS

This work is supported by the Office of Naval Research through the Advanced Materials Intelligent Processing Center program.

CRYSTAL MELTING MODEL

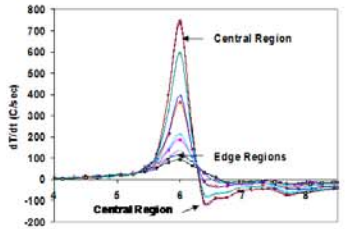
MAFFEZZOLI, KENNY, & NICOLAIS (1989)



$$X_f = \frac{1}{Q_T}\int_0^t \frac{dQ}{dt}dt$$ Q_T: Total Heat of Fusion $K = K_o \exp\left(\frac{-E_a}{RT}\right)$ K_o: Pre-exponential Factor, E_a: Activation Energy

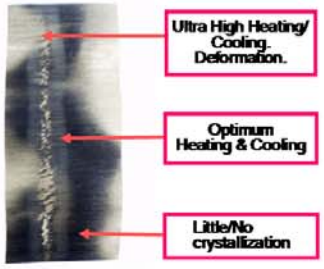
$$\frac{dX_f}{dt} = K\left(1 - X_f\right)^n$$ Simple n^{th} Order Kinetic $X_w = X_{wm}\left(1 - X_f\right)$ Degree of Crystallinity

EXPERIMENTAL RESULTS FOR HEATING/COOLING RATES

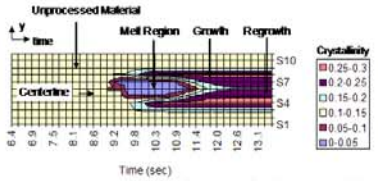


Heating rates in excess of 700°C/sec
Cooling rates of 150°C.

REGIONS OF CRYSTALLINITY

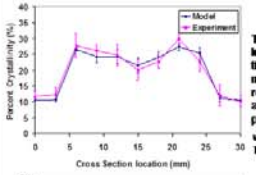


MODEL PREDICTIONS VERSUS EXPERIMENTAL RESULTS



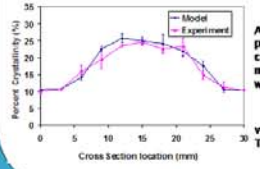
- Three specific regions of crystallinity develop within the prepreg with the single nozzle experiment
- In the outer edges, little crystallinity is developed, as the temperatures in these areas are not high enough to increase crystallinity.
- Closer to the centerline, the crystallinity is found to increase rapidly as the conditions are ideal for crystal growth without melting. The crystallinity will not reach equilibrium state, as the residence time at such temperatures is very short.
- The center section will undergo both melting and recrystallization and reaches a percentage of crystallinity that is slightly less than that achieved on the side regions.
- The highest heating and cooling rates are along the mid section and will also contribute to a lower crystallinity in the center region.

MODEL PREDICTIONS VERSUS EXPERIMENTAL RESULTS



The central region has a lower crystallinity due to the fact that it undergoes melting and recrystallization which agrees with model predictions.

v=25mm/s, h = 10mm, T_{gas} = 850°C



At higher velocities the prepreg only undergoes crystallization without melting which also agrees with model predictions.

v=75mm/s, h = 10mm, Tgas = 850°C