

# The influence of temperature and strain rate on the constitutive and damage responses of polychlorotrifluoroethylene (PCTFE, Kel-F 81)

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## Abstract

Polychlorotrifluoroethylene (PCTFE), also known as Kel-F 81, is a semi-crystalline fluoropolymer. Although it has been employed in a wide range of cryogenic components, valve seats, seals, and microelectronics packaging, its mechanical behavior has received limited coverage in the literature. In this work, we present the tensile and compressive constitutive response of PCTFE for a range of temperatures (−85 to 150 °C) and strain rates ( $1 \times 10^{-4}$ – $2.9 \times 10^3$  s<sup>−1</sup>). Both large-strain experiments based on flow stress and small-strain dynamic mechanical analysis (DMA) using the elastic modulus exhibit a strong increase in the glass transition temperature,  $T_g$ , with increasing strain rate. The quasistatic fracture behavior of PCTFE is presented using *J*-integral fracture experiments. Finally, a discussion of the implication of the constitutive and damage responses of PCTFE on impact failure modes observed in Taylor impact experiments is presented.

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## 1. Introduction

Polychlorotrifluoroethylene (PCTFE) is a fluoropolymer closely related in chemical structure to the more common polytetrafluoroethylene (PTFE) and shares many similarities in ductile mechanical characteristics. Notably, however, the replacement of one out of every four fluorine atoms along the carbon backbone with a larger chlorine atom prevents the crystalline phase transitions that dominate the mechanical response of PTFE (see Refs. [1–14]). A common trade name for PCTFE as manufactured by the 3M company is Kel-F 81. It is melt processable, resistant to most common chemicals, highly electrically insulating and extremely impervious to water absorption. The most common industrial uses are for cryogenic components, valve seats, seals, and microelectronics

packaging. As such, while the literature contains numerous studies on adhesive properties of PCTFE (see for example Refs. [15–19]), there is limited information on the mechanical or failure behavior of bulk PCTFE. McCrum [20,21] performed early dynamic mechanical analysis (DMA) studies to investigate the effects of temperature and crystallinity on the small-strain behavior of PCTFE, and a limited number of authors have studied the effect of temperature on the tensile response of PCTFE [22–25]. A brief study of PCTFE failure surfaces employing low resolution scanning electron microscopy has been presented by Shoemaker and Sterling [26]. The crystal structure is reported by Mencik [27] and the glass transition temperature is investigated by Hoffman ( $T_g = 52$  °C), Privalko ( $T_g = 64$  °C), Khanna ( $T_g = 75$  °C), and Chang ( $T_g = 47$ – $77$  °C depending on crystallinity) [28–31]. The shock response of PCTFE has been reported by Marsh, Anderson and Sheffield [32–34]. The pressure and temperature dependences of the dielectric and ultrasonic properties of PCTFE have been detailed by Samara and Fritz [35].

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A motivation for this research was to investigate the relationship between this material and a copolymer of PCTFE and polyvinylidene fluoride (PVDF) called Kel-F 800. The copolymer was also manufactured by the 3M company, consisting of 75 mol% PCTFE and 25 mol% PVDF. However, the introduction of PVDF into the copolymer dramatically changes the crystallinity, the glass transition temperature, and the mechanical response. As such, direct comparison between Kel-F 81 and Kel-F 800 offers limited insight. An extensive investigation of the influence of temperature and strain rate on the constitutive and damage responses of Kel-F 800 will be presented in a subsequent publication. Nevertheless, the current investigation of PCTFE presents a number of unique features of importance to the mechanical response of the homopolymer.

In the current work the constitutive response of PCTFE is presented in both tension and compression to large strains. The compressive response is presented for a temperature ranging from  $-85$  to  $150$  °C and strain rates of  $1 \times 10^{-4}$ – $2.9 \times 10^3$  s $^{-1}$ . The tensile response is reported for a temperature ranging from  $-50$  to  $100$  °C and strain rates of  $1.1 \times 10^{-4}$ – $1.1 \times 10^{-1}$  s $^{-1}$ . Yield behavior in tension is coupled with necking in the gage section. At temperatures of  $23$  °C and above the neck is subsequently drawn out, whereas at lower temperature the neck localizes leading to the prompt failure. Both the large-strain experiments based on flow stress and small-strain dynamic mechanical analysis (DMA) using the elastic modulus exhibit a strong increase in the glass transition temperature,  $T_g$ , with increasing strain rate. The quasistatic fracture behavior of PCTFE is presented using  $J$ -integral fracture experiments. Below  $T_g$ , the fracture toughness of PCTFE has a relatively low temperature dependence, but above  $T_g$  the increased ductility results in a significant increase in  $J_{IC}$  around  $65$  °C followed by a precipitous drop in toughness. A discussion of the implication of the constitutive and damage responses of PCTFE on impact failure modes observed in Taylor impact experiments is presented.

## 2. Experimental procedure

### 2.1. Material and sample preparation

A PCTFE billet of compression molded Neoflon<sup>®</sup> measuring  $500 \times 500 \times 25$  mm<sup>3</sup> was purchased for investigation in this study. The molding powder used is manufactured by Daikin Industries of Japan. The crystallinity was estimated from the differential scanning calorimetry (DSC) melt endotherm obtained in a TA Instruments Q1000, Fig. 1. Measurement of the melt peak in PCTFE determined a release of  $\Delta H_f^{\text{sample}}$  equal to  $19.9$  J g $^{-1}$ . Crystallinity was calculated as  $X_C = \Delta H_f^{\text{sample}} / \Delta H_f^0$  using the literature value for  $\Delta H_f^0$  of  $43.5$  J g $^{-1}$  [36], which suggests a crystallinity of 46%. Other references suggested slightly different value for  $\Delta H_f^0$  of  $43.2$  J g $^{-1}$  [37] and  $41.9$  J g $^{-1}$  [38]. The use of both of these slightly lower estimates would result in higher crystallinity estimates. The density was measured by helium pycnometry and

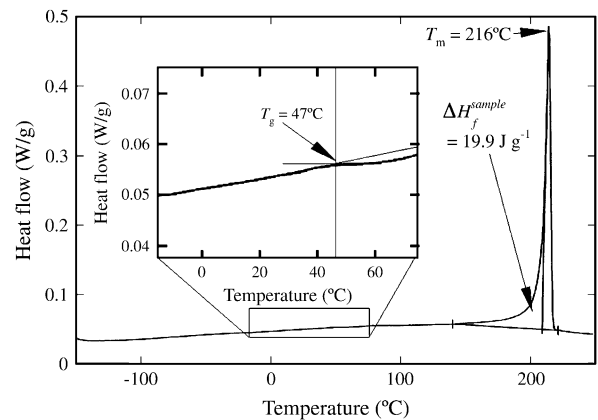


Fig. 1. DSC scan of PCTFE.

was found to be  $2158 \pm 1$  kg m $^{-3}$ . Following the equation proposed by Hoffman and Weeks [28] this implies a crystallinity of 75%.<sup>1</sup> Different methods of measuring crystallinity have been compared by Murthy et al. [39]. The glass transition temperature,  $T_g$ , was measured to be  $47$  °C, from a very subtle endotherm in the DSC trace. The DSC melt peak temperature was  $216$  °C at  $10$  °C min $^{-1}$ . Specimens were machined from the pressed and sintered billets of pedigreed PCTFE while ensuring a nominal temperature rise to prevent changes in the material's crystallinity.

### 2.2. Compression and tension

Given the ductile nature of PCTFE, large-strain deformations were investigated. For this reason, all strains referenced in this paper, unless otherwise noted, are true-strains (logarithmic strains). A constant true strain rate was maintained for all large-strain compression experiments. The feedback loop from the testing machines was closed to correctly slow the cross-head speed as the samples thinned. True-stress was calculated assuming a constant sample volume. The compression sample geometry chosen was  $6.375$  mm diameter by  $6.375$  mm long right-regular cylinders. The aspect ratio of 1:1 is smaller than the 1:1.5–1:2 values often employed in compression tests on metals, but the sample size and ratio were chosen to conserve material for the large number of tests required and prevent the trapezoidal shearing deformation mode observed in some soft polymers.

For the compression tests both MTS 880 and MTS 810 servo-hydraulic machines were utilized. These machines ran MTS TestStar software allowing for full control over the test

<sup>1</sup> As previously discussed for PTFE [2,3,5] density methods routinely report higher values of crystallinity than DSC. This is due to the partitioning by the density method of oriented domains but not thermodynamically crystalline domains of crystalline material, which is not included by DSC. Moreover, Hoffman and Weeks [28] used an immersion method to measure density as opposed to helium pycnometry. While the different methods generate different values for crystallinity, they are highly repeatable, provide meaningful comparison between studies as long as the technique for quantifying crystallinity is kept constant, and the different techniques similarly capture crystallinity changes due to thermal processing.

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