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Aging of poly(ether ether ketone) by heat and gamma rays — Its degradation mechanism and effects on mechanical, dielectric and thermal properties

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In radiation research facility, where heat and radiation are two crucial factors inducing material degradation, highly stable materials are required. In the present work, various properties of poly(ether ether ketone) (PEEK) exposed to heat and gamma rays in air are instrumentally investigated. If PEEK is aged by heat and gamma irradiation in air, chemical bond scission, oxidation, crosslinking, and char formation occur as competing mechanisms. The temperature plays a decisive role in degradation, while the irradiation accelerates it. The mechanical properties deteriorate much earlier than the dielectric properties with the progress of aging. This means that the mechanical properties can be an important condition monitoring factor compared to the dielectric properties for PEEK-insulated apparatus or cables. If PEEK is aged severely, its electrical conductivity increases dramatically, whereas both real and imaginary parts of complex dielectric permittivity decrease, showing a marked contrast to many industrially important polymeric insulating materials.

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1. Introduction

It is estimated that the capacity of electric power generation in the world reached about 5.95 TW in 2013. Fossil fuels are still dominant and they account for up to 82% of the total capacity [1]. Unfortunately, thermal power generation of electricity of 1 kWh using fossil fuels produces carbon dioxide more than 1 kg [2], which may cause serious environmental problems such as China's serious haze. According to a scenario of the International Energy Agency, the global electric power generation will increase up to 10.7 TW or more in 2040. This means that a capacity of 4.75 TW will be added in a period from 2014 to 2040. In the same period, 2.45 TW will be needed to replace old plants [3]. Since hydropower and renewable energy sources are limited geographically, nuclear power is a compromise as a clean energy technology [4].

Electric wires and cables play an important role in the delivery of energy and information. More than 1000 km of electric wires and cables are used in a nuclear power plant (NPP) [5]. Electrical insulation of cables is in most cases maintained by polymeric materials, which are not robust in a nuclear environment. Degradation or aging of polymeric insulation of cables may lead to a fatal accident in a NPP or malfunction of its devices or machines. This has become a matter of serious concern, since more than three quarters of NPPs are over 25 years old [1]. As far as NPPs in Japan, electrical insulating materials used in safety-related cables have been unchanged since the first NPP. That is, flame-retardant crosslinked polyethylene, flame-retardant ethylene-propylene-diene rubber, and silicone rubber have been mainly used. It is therefore important to consider the possibility of using new materials with high overall performances [6–9].

Poly(oxy-1,4-phenyleneoxy-1,4-phenylelecarbonyl-1,4-phenylene), known widely as poly(ether ether ketone) or PEEK, is a typical aromatic polyketone with a structure shown in Fig. 1. It is an important engineering polymer with excellent mechanical strength, high chemical and biological stability, and superior anti-radiation and heat-resistant properties [10–14]. Therefore, PEEK is potentially usable in a variety of fields and applications such as

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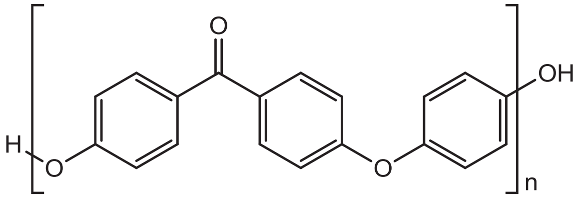

Fig. 1. Chemical structure of PEEK.

research facilities and aerospace, automotive, power and energy generation, and biomedical industries [14–22].

Regarding the degradation mechanism of PEEK, although much research has been conducted on thermal decomposition [23–27], long-term degradation has scarcely been studied. In the present paper, the mechanism and the effects of long-term degradation of PEEK caused by heat and gamma rays are discussed based on the results obtained by conducting various instrumental analyses.

2. Experimental materials and methods

2.1. Materials

Sheet-shaped PEEK samples with a thickness of about 1 mm were supplied by a Japanese company of manufacturing cables. They were aged thermally in air at various temperatures from 100 to 290 °C with or without concurrent irradiation of ^{60}Co gamma rays, as listed in Table 1. In the sample name, the letter H denotes that the sample was aged only thermally, while HR means simultaneous aging with heat and gamma rays. The numeral that follows H or HR represents the aging temperature.

2.2. Instrumental analyses

For these sheet samples, ultraviolet-visible (UV-Vis) spectra in an attenuated total reflection (ATR) mode were measured in the wavelength range from 360 to 800 nm using a Shimadzu UV-3100 PC spectrometer, while spectra of X-ray photoelectron spectroscopy (XPS) were measured using a JEOL JPS-9100 TR spectrometer mainly paying attention to the peaks assigned to oxygen and carbon. Further, Fourier transform infrared (FT-IR) absorption spectra were obtained in the wavenumber range from 500 to 4000 cm^{-1} at every 2 cm^{-1} in an ATR mode using a JASCO FT/IR-4200 spectrometer.

In addition, solid-state ^{13}C nuclear magnetic resonance (NMR) spectra were obtained for samples in the shape of chips using a JEOL JNM-ECA 400 spectrometer to evaluate possible changes of the skeletal structure of PEEK. Electron paramagnetic resonance (EPR) spectra were obtained for similar chips using a JEOL JES-FA 300 spectrometer in the magnetic field range from 310 to 320 mT at a microwave frequency of 10 GHz.

Thermal properties such as enthalpy of fusion, melting point of crystalline parts, and thermal decomposition temperature were measured either by differential scanning calorimetry (DSC) in the range from room temperature to 400 °C in dry nitrogen at a heating rate of 10 °C/min by a calorimeter (3100, Netzsch Japan) or thermogravimetric/differential thermal analysis (TG/DTA) from 35 to 600 °C in dry nitrogen by an analyzer (6200, Seiko Instruments). Thermal diffusivity was measured by a laser flash method (LFA, Netzsch LFA 447) according to ASTM E1461. For these thermal analyses, the sample shape was modified appropriately.

Table 1
Degradation conditions of sheet-shaped PEEK.

Degradation condition	Sample					
	HR100	HR165	HR200	H200	H265	H290
Temperature (°C)	100	165	200	200	265	290
Dose rate (Gy/h)	150	150	150	–	–	–
Aging time (h)	900	650	425	425	1025	550
	1800	1300	850	850	2050	1100
	2700	1950	1275	1275	3075	1550
		2520	1661	1661		2176

2.3. Mechanical and dielectric properties

To evaluate mechanical performances of PEEK, elongation at break (EAB) and tensile strength were tested using a dumbbell-shaped test piece by a universal testing machine (5565, Instron) at a tensile speed of 500 mm/min according to the Japanese Industrial Standard JIS K 7161: 2010.

As an important property of dielectric behavior, dc conductivity was measured by applying a dc voltage of 5 kV to a sample sheet of about 1.0 mm thick at 30 °C. Here, the conductivity was calculated using the dc current value measured 60 min after the start of voltage application, at which the current became almost constant, using an ADCMT 8252 electrometer (ADC). Complex dielectric permittivity was also measured for a similar sample sheet in the wide frequency range from 10^{-2}–10^{5} Hz at temperatures from 25 to 300 °C in vacuum using a Solartron SI126096W impedance analyzer using two circular electrodes with a diameter of 20 mm.

3. Results and discussion

3.1. Aging mechanism of PEEK exposed to heat and gamma rays

Several characteristic peaks can be recognized in FT-IR spectra shown in Fig. 2. Note that a logarithmic scale is used as the abscissa so that "fingerprint spectra" in the wavenumber range below 2000 cm^{-1} can be shown clearly. In addition, each spectrum is shifted vertically by 0.5 to increase the visibility. Although the aging at 200 °C hardly changes the FT-IR spectra regardless of the presence or absence of concurrent irradiation of gamma rays, the spectra are obviously changed by the aging at 290 °C. Namely, after the aging at 290 °C, the absorbance decreases dramatically except for the two oxidation-related peaks at 1730 and 1100 cm^{-1}. This is attributable to the scission of C—H bonds and the following

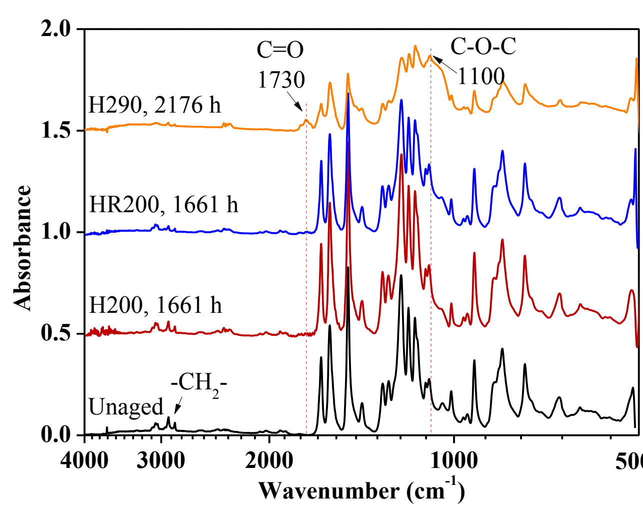

Fig. 2. FT-IR spectra of PEEK samples aged under different conditions. Each spectrum is shifted vertically by 0.5.

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