Temperature-mediated Microstructure Evolution and Densification Mechanism of F2314 Fluoropolymer Binder during Warm Isostatic Pressing

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1. Characterization methods

The morphology of the raw powder was observed using a SEM 5000 microscope (Guoyi Quantum Technology Co., Ltd.) at an accelerating voltage of 10 kV. Prior to observation, the sample surfaces were sputter-coated with gold for 60 s. In addition, the particle size distribution was statistically analyzed using Image J software.

The F2314 samples formed by isostatic pressing were machined into Φ5 mm × 5 mm specimens. The deformation of the samples during heating was measured using a TMA 402 F3 thermomechanical analyzer (NETZSCH, Germany). The test temperature ranged from 40 to 150 °C at a heating rate of 5 °C/min under a static load of 0.1 N, with a nitrogen atmosphere flow rate of 20 ml/min.

The F2314 raw powder was heated to various temperatures for in-situ FTIR analysis using a Fourier transform infrared spectrometer (Nicolet Is-50, Thermo Fisher Scientific, USA) to monitor the variation of its chemical bonds during preheating. Additionally, thin slices were cut from the isostatically pressed F2314 material for FTIR testing at room temperature. All spectra were collected in ATR mode with a resolution of 4 cm^{-1}, 32 scans, over the range of 4000–550 cm^{-1}.

2. Result

2.1 Raw material morphology

As shown in Fig. S1(a) and (b), at a magnification of 200×, the F2314 raw powder exhibits an irregular morphology, with a D50 of 34.0 ± 15.6 μm, indicating a broad size distribution. When observed at 1000× magnification [Fig. S1(c)], the powder surface displays a rough and porous structure. Further magnification to 5000× [Fig. S1(d)] reveals that the particles are densely packed by microspheres with a uniform size of approximately 0.42 μm. This morphological feature is closely related to the polymerization method of F2314.

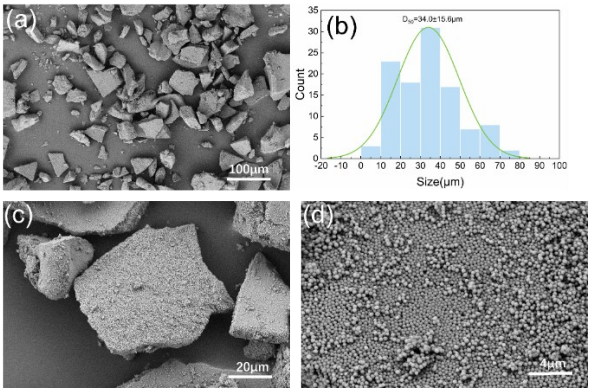

Fig. S1 SEM images of the raw material and the particle size distribution of F2314.

2.2 Thermomechanical Analysis

As shown in Fig. S2, during the pressing of F2314 samples, the densification and thermal expansion deformation vary nonlinearly with temperature (65°C to 140°C), influenced by porosity, chain mobility, and residual gas expansion. At 65°C, abundant pores allow chain movement and air expansion to promote deformation, while pore shape adjustment partly counteracts it. At 80°C, reduced porosity improves continuity and chain mobility, weakening pore restraint and increasing expansion. Above 95°C, densification exceeds 99% and pores close, making expansion behavior intrinsic. High density suppresses gas-induced expansion, minimizing overall thermal deformation.

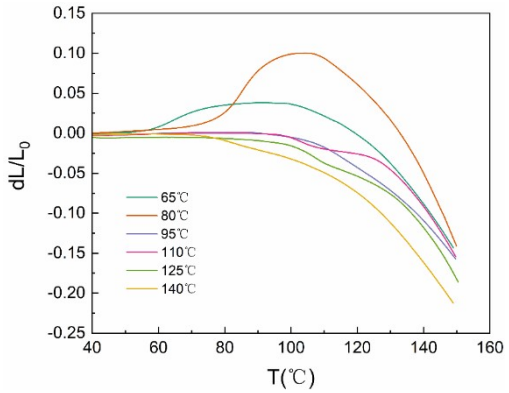

Fig. S2 TMA profiles of F2314 compacted at various compaction temperatures

2.3 Infrared spectroscopy analysis

As shown in Fig. S3(a), no new chemical bonds were observed in the F2314 raw material when heated from 65°C to 140°C, indicating that no degradation or cross-linking of polymer chains occurred during the heating process. The main absorption peaks correspond to the stretching vibrations of -CCl (956 cm^{-1}), -CF_2- (1115 cm^{-1}), and -CF- (1151 cm^{-1}), as well as the bending vibrations of -CH_2- (1384 and 1427 cm^{-1}).[22,25] Fig. S3(b) shows that the infrared spectra of the F2314 samples formed by isostatic pressing at the same temperatures exhibited no significant changes, demonstrating that the material remains chemically stable under combined temperature and pressure conditions.

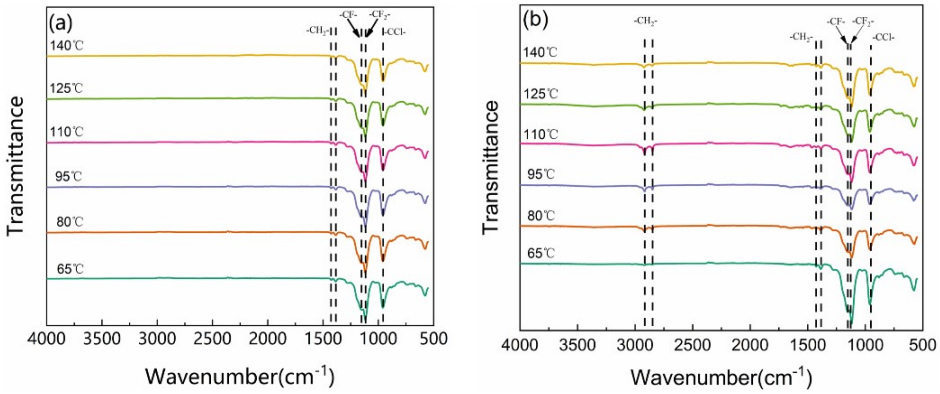

Fig. S3 FTIR spectra of F2314 (a) heated and (b) compacted at various temperatures.