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STAGE-RESOLVED SURFACE AND GAS-PHASE EVOLUTION OF OIL-IMPREGNATED INSULATION PAPER UNDER PARTIAL-DISCHARGE DAMAGE

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Outline

- Introduction & motivation
- Materials and test model (CIGRE Method II)
- Test conditions and damage staging
- Table 1 — five stages of PD damage
- Results: surface morphology and roughness evolution
- Results: chemical composition (FTIR / XPS)
- Results: cavity gas composition (GC-MS)
- Proposed mechanism
- Conclusions

1. Introduction

- Reliability of oil-barrier insulation determines the safety of power-transformer operation
- Manufacturing / in-service defects (bubbles, delaminations, inclusions) → local field concentration → partial discharges (PDs) in residual gas cavities
- PD charged particles carry several–tens of eV, exceeding the cellulose bond energy (a few eV) → macromolecular chain rupture
- Local heating within the discharge channel (up to ~1000 °C) triggers thermal decomposition
- Prior work treated thermal ageing, dissolved-gas diagnostics, surface chemistry and PRPD classification as separate problems
- Need: a joint, time-resolved picture linking PRPD pattern ↔ morphology ↔ chemistry

Aim of the Study

- Establish a stage-resolved description of partial-discharge damage in a flat gas-filled defect in oil-impregnated kraft paper
- Jointly analyse:
  - PRPD patterns
  - Optical and AFM morphology
  - Surface functional groups (FTIR, XPS)
  - Cavity-gas composition (GC-MS)
- Identify the physical origin of the observed damage stages
- Assess the potential diagnostic value of their coupled signatures

2.1 Materials & Test Model

- Mineral transformer oil (grade 25#) as liquid dielectric; electrical-grade kraft paper (grade JZ-70) as solid insulation
- Vacuum-dried and vacuum-impregnated with oil, reproducing the real manufacturing cycle
- Test model built per CIGRE Method II: three-layer sandwich
  - Top / bottom layers: 1-mm insulation pressboard
  - Middle layer: 0.125-mm insulation paper with a 38-mm central hole → flat internal gas cavity
- Sealed with silicone sealant; assembly immersed in an acrylic vessel filled with mineral oil
- Parallel models fitted with a glass indicator tube (silicone oil) to track cavity gas-volume changes and enable periodic gas sampling

2.2 Test Conditions & Damage Staging

- 10 kV power-frequency AC (mean cavity PD inception voltage 7.5 kV); 70 °C ageing-chamber temperature (IEC/IEEE class-A maximum continuous operating temperature)
- PD signals captured with a digital oscilloscope (100 MS/s, 600 cycles/acquisition) through an RC detection impedance
- Acquisitions every 5 min during the first 5 h, then every 5 h, over ~500 h to breakdown
- Joint analysis of PRPD grey-scale pattern, surface morphology and cavity-gas composition → five distinguishable stages (Table 1)
- Stage transitions identified qualitatively (simultaneous change across all three feature groups), not by a fixed numerical threshold

Table 1 — Five Stages of PD Damage

Stage	Approx. duration	PRPD pattern shape	Surface morphology	Dominant discharge type / cavity gas
I	~1.5 h	"turtle-back"	fibrous, rough	pulse-type; high electronegative-gas concentration
II	~50 h	"band"	fibre burn-off, smoothing	pseudo-glow discharge; O2 depleted
III	~120 h	"rabbit" / periodic "cluster"	fibre delamination, liquid droplets	pulse-type; CO2, H2O, hydrocarbons rise
IV	~280 h	"band"	fibre splitting, crystalline solid	pseudo-glow discharge; CO2, H2O and hydrocarbons decreased
V	→ breakdown	"triangle" / "cone"	pits, electrical treeing	pulse-type; CO2, C2H2 rise again

Five stages identified consistently across PRPD pattern, surface morphology and cavity-gas composition (original experimental data).

3.1 Surface Morphology & Roughness Evolution

- Stage I: disordered fibrous surface
- Stage II: fibres progressively burned off by charged-particle bombardment → surface smooths, roughness Sa decreases
- Stage III: fibres delaminate; liquid droplets (oxidation product) accumulate on the surface
- Stage IV: fibres crack and split; crystalline solid forms → sharp protrusions increase roughness
- Stage V: pits develop around solid deposits → dendritic electrical trees → breakdown channel
- Roughness is non-monotonic: decreases in stages I–II, then increases from stage III onward, slowing near breakdown

AFM: Nanoscale Observations

- AFM compares ordered vs. amorphous surface regions using three concentric zones, three sectors per zone
- Ordered regions are damaged earlier and more markedly: uniformly distributed chain scissions already visible by 175 h
- Amorphous regions: comparable degradation appears later, and is more gradual and patchy
- Possible reason: dense packing and an extensive H-bond network reduce conformational freedom — ordered chains are more brittle under charged-particle impact; disordered chains can partially relax the deposited energy
- Caveat: AFM probes only the outermost nanometre-scale layer — not directly extrapolable to the bulk structural response

3.2 Chemical Composition — Surface Products

- FTIR: C=O band (1690–1760 cm^{-1}) with aldehyde bands (2720, 2820 cm^{-1}) appear at stage III
- After acetone cleaning: aldehyde bands disappear, an attenuated C=O band persists → droplets are mainly carbonyl compounds; part of the C=O forms directly within the cellulose chain
- Stage IV solid product: C=O without an aldehyde component → carboxylic acids dominate
- XPS C 1s: O–C=O (carboxyl) first appears at stage III; weak C–N (amine, stage IV) and C–ONO_2 (nitro, by 385 h)
- C–C/C–H rises 26.9% → 68.9%; C–O falls 46.6% → 14.5%; C=O/O–C–O falls 26.5% → 9.8% — progressive carbonisation with partial oxidation

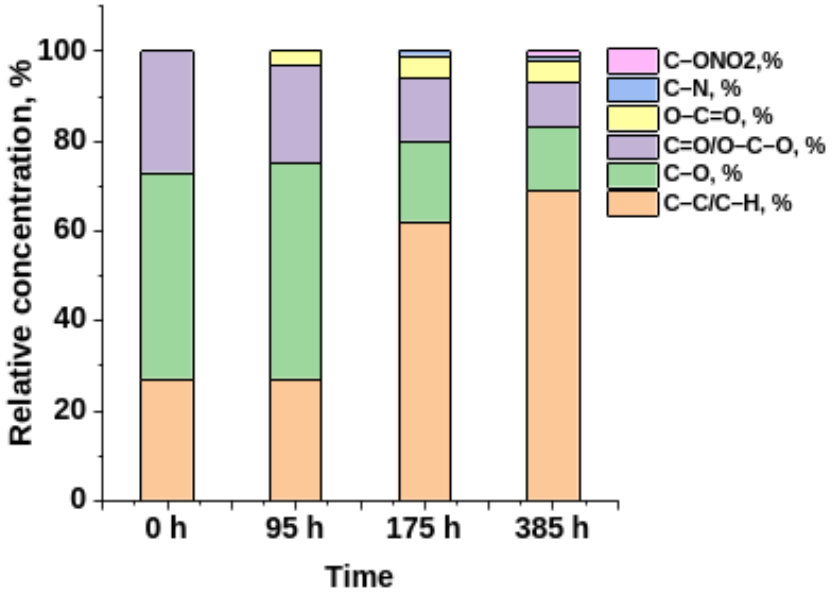


Fig. 1. Evolution of surface carbon functional groups (C 1s XPS).

Cavity Gas Composition (GC–MS)

- Initial air charge (20.95% O_2) almost completely consumed by 10 h (→ 0.02%)
- Stage III: CO_2, H_2O and hydrocarbon gases (CH_4, C_2H_2, C_2H_6, H_2) rise sharply — 6.9%, 2.9% and 39.1%
- Stage IV: these components decrease
- Stage V: they rise sharply again — CO_2 to 11.0%, hydrocarbons to 51.0%
- Alternation of gas depletion/enrichment tracks the dominant discharge type: electronegative-gas-rich → pulse-type (stages I, III, V); depleted → weak pseudo-glow (stages II, IV)

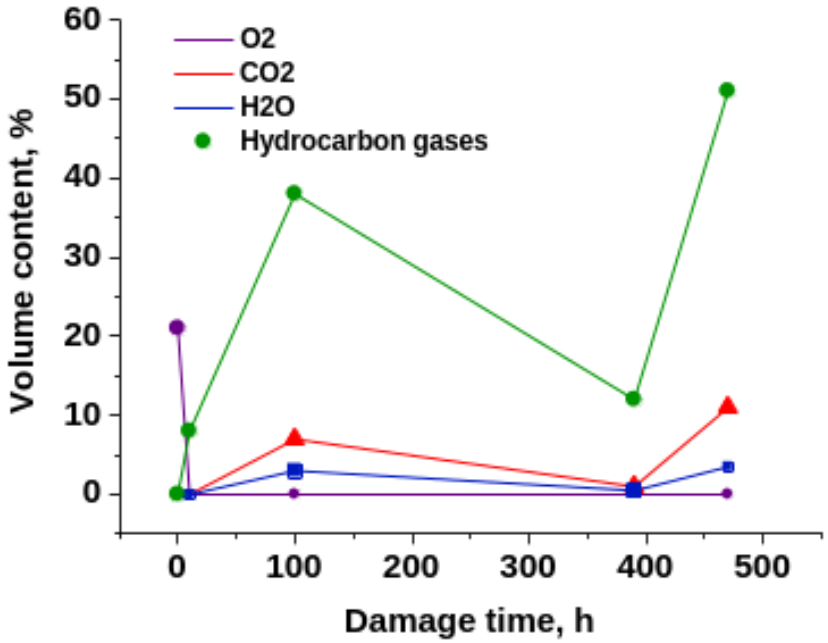


Fig. 2. Evolution of cavity gas composition (GC-MS).

Proposed Mechanism

- Charged-particle bombardment + local heating → cellulose covalent-bond scission → polymeric free radicals + low-molecular-weight hydrocarbon gases
- With oxygen / atomic oxygen / ozone → oxidation to oxygen-containing groups: –OH, –O–, C=O, –CHO, –COOH
- With reactive nitrogen species → amino- and nitro-groups
- Discharge intensity correlates with product state: intense pulse-type discharge (stages I, III, V) → mainly liquid droplets; weak pseudo-glow discharge (stages II, IV) → crystalline solid
- Field enhancement around solid deposits is proposed as a precursor to pit formation and electrical treeing — a physically motivated interpretation, not directly measured in this study

4. Conclusions

- Five distinguishable stages identified from the simultaneous evolution of PRPD pattern, surface morphology and cavity-gas composition
- Fibre burn-off initially smooths the surface; later accumulation of liquid/solid products, pit formation and tree growth increase roughness; AFM shows earlier, more pronounced damage in ordered regions
- Cellulose surface becomes progressively carbon-rich (C–C/C–H: 26.9%→68.9%; C–O: 46.6%→14.5%); carbonyl, carboxyl, amine and nitro groups document concurrent oxidation and nitrogen fixation
- O_2 depletion and non-monotonic CO_2/H_2O/hydrocarbons accompany alternation between pulse-type and pseudo-glow discharge
- Breakdown is linked to field enhancement around solid deposits, followed by pit development and electrical-tree growth
- Future work: quantify PRPD-descriptor correlations; test reproducibility across cavity sizes, voltages, temperatures and moisture contents