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Compression-Induced Deformation and Gas Permeability of Graphite Foil Under Stress Relaxation: Experimental Study and Modeling

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Abstract

Graphite foil is widely used as a sealing material in flange joints in the form of gaskets or gasket components. Predicting gasket permeability during stress relaxation remains challenging because both the compression state and the gas pressure affect leakage. No unified semi-empirical model based on the Darcy–Klinkenberg framework with compression pressure as a direct input has been available for use in flange-joint numerical simulations. Graphite foil gaskets with a density of about 1.0 g/cm³ and a thickness of ~1.5 mm were tested under compression pressures from 5 to 100 MPa. Helium leakage was measured at helium pressures from 0.5 to 8 MPa. Leakage and deformation during loading and unloading were recorded using EN 13555-based procedures. The results were analyzed using a Darcy–Klinkenberg formulation and equivalent slit- and capillary-based representations of the leakage channels. The second-order model reproduced the pressure-dependent leakage more accurately than the first-order Darcy approximation ($R^2 \geq 0.9985$ vs. 0.916–0.992), particularly where slip-flow effects were significant. Exponential dependences of the intrinsic permeability and the Klinkenberg coefficient on deformation and power-law relations with compression pressure are proposed to model leakage during unloading. The proposed semi-empirical model allows estimation of graphite-foil permeability under stress relaxation with the use of EN 13555 test procedures and its subsequent implementation in numerical simulations of flange joints. Limits of the model's applicability, including loading regime, ranges of compression pressure, gas pressure and anisotropic nature of permeability, are discussed.

Keywords: graphite foil; gas permeability; stress relaxation; Klinkenberg model; compressive deformation; flange gasket

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1. Introduction

Graphite foil (GF) made from exfoliated graphite is a flexible carbon material that is widely applied in industry [1]. Its primary use is as an elastic sealing component in bolted flange joints of equipment and pipelines in the oil-and-gas industry and in thermal and nuclear power generation [2,3]. Exfoliated graphite retains chemical resistance, elasticity and strength over a wide range of temperatures and pressures, withstands chemical and radiation exposure, and—being a porous material—remains permeable to fluids even under strong compression [4]. The shape and size of pores under compression along and perpendicular to the rolling plane of graphite foil were studied up to 100 MPa by small-

angle neutron scattering and mercury porosimetry [5,6]. Pores were shown to be ellipsoidal and to shrink from about 100 nm to about 4 nm upon compression. Under perpendicular compression, large pores collapse into smaller pores, and the pore size increases when the load is released. Because low permeability is a key requirement for sealing materials, effective sealing normally requires compression of exfoliated graphite at 10–150 MPa [7].

In current practice, leak-tightness calculations of flange joints under relaxation rely on experimentally measured gasket permeability as a function of surface compression pressure and on gasket relaxation parameters obtained according to EN 13555 [8] and the ASTM/ROTT procedure [9]; the corresponding gasket constants are then used in flange-joint analysis. The experiments are performed on standard-size specimens under the assumption that geometric size factors do not affect gasket compression, which does not fully represent the behavior of real bolted joints with varying dimensions. Under transient operating conditions, in particular during heating and cooling, non-uniform thermal expansion of the flange components can lead to substantial changes in the surface compression on the gasket, which is itself non-uniformly distributed because of complex flange deformations during service [10] and external loads [11]. It is therefore important to derive dimensionless models of gas permeability versus medium pressure and compression that can be used in flange-joint analysis under complex loading and subsequently coupled with transient thermomechanical simulations.

Several research groups have demonstrated the feasibility of effective permeability models for exfoliated-graphite-based materials. Bramsiepe et al. [12] proposed a capillary model that predicts the permeation of volatile fluids through graphite foil as a function of medium pressure and compression and linked it to the total porosity computed from geometrical considerations.

Researchers from the École Polytechnique de Montréal developed the laminar-molecular flow (LMF) model and validated its applicability to flange and packing seals over a range of compression pressures and media [13–15]. They showed that, for graphite-foil-based materials, a second-order capillary model describes the dependence of permeability on medium pressure more accurately than the diffusion model or the Ergun model [16]. They also reported nearly linear dependences of the effective capillary diameter and slit height on compression pressure at low compression (up to about 40 MPa), with substantial deviations from linearity at higher pressures [17]. The characteristic size of the leakage channels was on the order of tens of nanometers. In the work of Marchand et al. [13] and Jolly and Marchand [18] the applicability of the model for various gases was investigated and confirmed for helium, argon, methane and liquids, both for graphite foil and compressed fiber sheets. In earlier work, we investigated the pore structure of graphite foils prepared by different routes and computed parameters of the capillary model [19,20]. The characteristic pore sizes inferred from mercury porosimetry and nitrogen adsorption were somewhat larger than the characteristic sizes of the permeability channels, which is consistent with a presence of locked channels and bottle-neck topology of the leakage paths [19,20].

From the application point of view, an important task is to construct a model of the dependence of graphite-foil permeability on compression pressure during stress relaxation. The influence of geometric size factors on the permeability versus compression and medium pressure dependences requires an analytical approach to gasket modeling in flange joints; such models have been successfully developed in a number of studies [21–23]. Kobayashi proposed an empirical model based on an exponential dependence of permeability on strain [24] while Gu and co-workers used power-law functions to describe the dependence of permeability on capillary diameter [25]. Jolly et al. [18] and Kazeminia

et al. [26] have found exponential leakage strain relations for graphite gaskets and packings respectively. Bouzid [27] and Volgin [28] used Kriging and Bezier-surface approximations, respectively, to analyze unloading permeability from BWT and EN 13555 data.

Graphite foil is known to be an elasto-plastic material, and its deformation under load reduction is nonlinear [29]. Empirical and semi-empirical stress–strain models have also proven effective in related structural applications [30,31], further motivating a similar approach for graphite-foil gaskets. It was previously shown that the dependence of leakage rate on compression pressure for gaskets during relaxation can be obtained using a strain-mapping approach, with a power-law dependence of leakage on strain and an exponential dependence of compression pressure on strain [18,26]. This is consistent with the exponential dependence of compression pressure on strain and density that is typically observed for compressed graphite foil [32,33].

Despite these advances, a model that directly connects the Darcy–Klinkenberg permeability parameters to compression pressure via standard EN 13555 test data—and is therefore immediately applicable to numerical simulation of flange joints under stress relaxation—has not been reported. Existing approaches either rely on tabulated permeability data at discrete compression levels without an analytical functional form or use empirical correlations that are not linked to the microstructural parameters (channel size and count) of the Darcy–Klinkenberg framework. The present work addresses this gap by: (i) fitting the second-order Darcy–Klinkenberg model to EN 13555 leakage data across six compression levels; (ii) establishing exponential $K_{\infty}(\epsilon)$ and $B(\epsilon)$ dependences supported by physical reasoning; and (iii) eliminating strain via the compression-pressure–strain relation to produce a closed-form model $G(\sigma, \Pi)$ calibrated directly from standard test data. The input data are EN 13555-based measurements of helium permeability and deformation during stress relaxation after compression in the range of 5–100 MPa in order to ensure the practical applicability of the model to standard test data. The results allow us to verify a two-dimensional permeability model of graphite foil in a plane-stress formulation with consistent mechanical characteristics, which can be subsequently used to simulate the behavior of materials in flange gasket designs under loss of compression force during real service.

2. Materials and Methods

2.1. Preparation of Graphite Foil Specimens

Natural flake crystalline graphite of grade GT-1 (Tayginskoye deposit, Tayginsky GOK LLC, Kyshtym, Russia) with an ash content of 4–6% and a particle size of approximately 250 μm was used as the starting material for exfoliated graphite. The natural graphite was purified by impregnation with a 40 wt.% aqueous solution of sodium hydroxide, heated to 350 $^{\circ}\text{C}$ and, after cooling, neutralized with diluted nitric acid and washed with distilled water. The resulting flake graphite had an ash content of 0.2–0.5% and a particle size of 250 μm .

The purified graphite was treated with 98% nitric acid at a graphite-to-acid mass ratio of 1:1.4. The resulting graphite nitrate was rinsed with water at an acid-to-graphite ratio of 1:5 on a porous glass filter and then dried on the filter at 60 $^{\circ}\text{C}$ for 6 h. X-ray diffraction showed that the product corresponded to stage 2–3 graphite intercalation compounds. The XRD pattern is provided as Supplementary Figure S1. The characteristic 00l reflections are consistent with a stage 2–3 intercalation structure [34,35].

Exfoliated graphite was produced by thermal shock in a tubular furnace at 900 $^{\circ}\text{C}$ under an air flow.

Graphite foil (GF) specimens were prepared by uniaxial pressing of exfoliated graphite in a die to nominal dimensions of approximately 160 $\times$ 160 mm in the in-plane direction. The target (nominal) density was approximately 1.0 g/cm³ and the target (nominal)

thickness was 1.5 mm; actual measured values for the specimens used in this study are given in Table 1. Annular test specimens (inner diameter 49 mm, outer diameter 92 mm) were cut from these sheets (see Supplementary Figure S2 for photographs of a representative sheet and a cut annular specimen).

Table 1. Physical and mechanical properties of the graphite-foil specimens.

Property	Value
Thickness, mm	1.53 ± 0.01
Density, g/cm ³	1.04 ± 0.02
Tensile strength Pmax, MPa	5.93 ± 0.10
Compressibility Ec, %	41.80 ± 0.30
Recovery Er, %	12.00 ± 0.30

2.2. Physical and Mechanical Characterization

Length and width were each measured three times per specimen side (in the middle and on both ends) using the 1 mm-scale ruler; the uncertainty of each dimension was taken as ±0.5 mm (half the least count). For a representative sheet of approximately 160 × 160 mm, this yields a relative uncertainty in the base areas of about 1.3%. Thickness was measured with a micrometer (precision 0.01 mm) at four points (one in each corner of the sheet) no closer than 30 mm from the edge; the arithmetic mean was taken as the result.

The density ρ (g/cm³) of the specimens was calculated as follows:

$$\rho = \frac{m}{H \times s} \quad (1)$$

where m is the specimen mass (g), H is the specimen thickness (cm) and s is the base area of the specimen (length × width, cm²).

Compressibility and recovery were measured on a PSP-type tester using an indenter of diameter 11.3 ± 0.06 mm (1 cm² area). Compressibility and recovery were measured on three specimens per batch. The indenter was lowered onto the specimen at least 10 mm from the edge; a preload of 75 ± 2 N was applied and held for 15 s, after which the preload thickness was recorded. A full load of 3500 ± 80 N was then applied for 60 s, and the thickness under full load was recorded. The load was then reduced back to the preload and held for 60 s; the recovered thickness was recorded.

Compressibility E_c (%) was calculated as follows:

$$E_c = \frac{(H_0 - H_1) \times 100\%}{H_0} \quad (2)$$

Recovery E_r (%) was calculated as follows:

$$E_r = \frac{(H_2 - H_1) \times 100}{H_0 - H_1} \quad (3)$$

where H_0 is the specimen thickness under the preload (mm), H_1 is the thickness under full load (mm), and H_2 is the recovered thickness (mm).

Tensile strength was determined on rectangular specimens 152.4 × 25.4 mm (per ASTM F152 procedure for flexible sheet gasket materials) punched from each selected sheet, and three specimens per batch were tested. The gauge length was 101.6 mm. The specimen was clamped in a tensile-test machine and stretched at 250 mm·min^{−1} until rupture, and the maximum applied force was recorded.

The tensile strength (MPa) was calculated as follows:

$$\sigma_{tens} = \frac{F}{S} \quad (4)$$

where F is the force at breakage under the load (N), and S is the cross-section of the sample (mm²).

2.3. Gas Permeability and Compression/Relaxation Tests

Leakage rate was measured on an AMTEC TEMES fl.ai1 (AMTEC Messtechnischer Service GmbH, Lauffen, Germany) gas-permeability test rig. Annular specimens with an inner diameter of 49 mm and an outer diameter of 92 mm were cut from graphite-foil sheets. Tests were carried out at room temperature (23 ± 3 °C). The ring was placed between flanges with raised faces of 88 mm outer diameter and 40 mm inner diameter, centered in the test chamber. A load corresponding to the prescribed compression pressure (5 to 100 MPa) was applied to the specimen, and helium was supplied to the inner cavity at a pressure of 0.5 to 8 MPa (absolute). The upper limit of 100 MPa was chosen so as not to exceed the compressive strength of the graphite foil. The outer perimeter of the gasket was sealed.

The test programs for measurement of the elastic modulus, gas-permeability dependence on medium pressure and compression-pressure dependence are summarized in Table 2. Specimen thickness was monitored with displacement sensors at three points around the gasket periphery. The leakage volume was measured over 90 min using both the pressure-rise method and helium leak detection, with monitoring of the pressure in the inner and outer chambers. The compression pressure was corrected for the contribution of the medium pressure. Elastic-property tests were performed using the same protocol without gas flow. The number of initial compression pressure levels follows the EN 13555 protocol and limits the number of points due to the long stabilization time (≈ 1.5 h per measurement).

Table 2. Test programs for gas permeability and compression/relaxation experiments.

Program	Description
1. Permeability vs. medium pressure at fixed compression	Permeability is measured sequentially at 5, 20, 40, 20 (after 40, unloaded to 20), 80, 100 MPa: $5 \rightarrow 20 \rightarrow 40 \rightarrow 20 \rightarrow 80 \rightarrow 100$ MPa. At each compression level the medium pressure is increased stepwise from 0.5 to 8 MPa; the test at each compression point lasts 1.5 h.
2. Gasket height (deformation) vs. compression pressure during loading and unloading	The procedure follows EN 13555 with extensions. The gasket is loaded to a target pressure and then unloaded to 10 MPa following the sequence: $10 \rightarrow 20 \rightarrow 10 \rightarrow 40 \rightarrow 10 \rightarrow 60 \rightarrow 10 \rightarrow 80 \rightarrow 10 \rightarrow 100 \rightarrow 10$ MPa. Flange-plate displacements were recorded during loading and unloading without medium pressure. Deformations measured at compression pressures above 10 MPa were used for the analysis in order to avoid the effects of surface unevenness and sensor calibration.
3. Permeability vs. compression pressure during loading and relaxation at fixed medium pressure	Helium permeability and deformation tests analogous to EN 13555 with helium at 4 MPa. The gasket is loaded to 20, 40, 60, 80 and 100 MPa and permeability is measured at each loading point. The compression pressure is then reduced to 5 MPa in steps: 2.5 MPa for 20 MPa; 5 MPa for 40 and 60 MPa; 10 MPa for 80 and 100 MPa. Permeability is measured at each step. The test at each point lasts 1.5 h.

2.4. Data Processing and Model Fitting

Experimental data were fitted with the equations introduced in Section 3 by nonlinear least-squares minimization using the Levenberg–Marquardt algorithm (LMA) with bound constraints imposed by the physical meaning of the parameters. Goodness of fit is reported as the coefficient of determination R^2 . Helium was used as the working gas in all permeability tests, and helium properties at $T = 298$ K were used in the model.

3. Results and Discussion

3.1. Physical and Mechanical Properties of GF

The principal physical and mechanical characteristics of the graphite-foil specimens are given in Table 1. The specimens exhibit typical values of strength and elastic properties for graphite foils of the chosen density and thickness. Morphology, pore-size distribution and the relationship between density, porosity and gas permeability of foils prepared by the same procedure were reported earlier [19,20].

3.2. Study of Permeability Dependence Versus Medium Pressure

For the graphite foils studied, helium permeability shows the characteristic dependence on medium pressure reported earlier (Figure 1). To fit these data, the model proposed by Grine and Bouzid [17] for flange gaskets was used: leakage channels are represented as a set of parallel slots or capillaries of identical size. For the slot geometry, the Navier–Stokes solution [36] can be written in first-order (Equation (5)) and second-order (Equation (6)) form as follows:

$$G = \frac{2MNh^3P_o^2}{3\mu RT \ln(r_o/r_i)} (\Pi^2 - 1) \quad (5)$$

$$G = \frac{2\pi MNh^3P_o^2}{3\mu RT \ln(r_o/r_i)} (\Pi^2 - 1) \left(1 + 12 Kn_o \frac{2-\Sigma}{\Sigma} \frac{1}{\Pi+1} \right) \quad (6)$$

where Σ is the gas-surface accommodation coefficient, which for most gases lies in the range 0.85–1 [36]. For helium, which does not chemisorb on graphite walls, $\Sigma = 1$ is assumed throughout. In what follows, the first-order model denotes the classical Darcy equation, and the second-order model includes the additional Darcy–Klinkenberg term in the Knudsen number; it describes the dependence of permeability on medium pressure over a wider range. Equations (5) and (6) can be written in an equivalent Darcy–Klinkenberg form by introducing the intrinsic permeability K_∞ and the Knudsen number. For a system of N parallel slots of height $2h$ and length L in a gasket of thickness H ,

$$K_\infty = \frac{2Nh^3}{3H} \quad (7)$$

The equivalent capillary representation (N cylindrical capillaries of diameter D and length L) gives

$$K_\infty = \frac{ND^4}{16LH} \quad (8)$$

For the slit model, the half-height h is taken as the characteristic channel size; the Knudsen number at normal conditions is then

$$Kn_o^{(slit)} = \frac{\lambda_o}{2h} \quad (9)$$

where λ_o is the helium mean free path at normal conditions. The Klinkenberg slip coefficient for the slit geometry is

$$b^{(slit)} = \frac{6\lambda_o}{h} = 12 Kn_o^{(slit)} \quad (10)$$

For the capillary model, the capillary radius $R = D/2$ is taken as the characteristic size, so that

$$Kn_o^{(cap)} = \frac{\lambda_o}{R} = \frac{2\lambda_o}{D} \quad (11)$$

$$b^{(cap)} = \frac{4\lambda_o}{R} = \frac{8\lambda_o}{D} = 4 Kn_o^{(cap)} \quad (12)$$

The numerical coefficients in front of Kn differ between slits and capillaries because of the different geometric definitions of the characteristic channel size. With these relations, Equations (5) and (6) become the Darcy–Klinkenberg representation:

$$G = \frac{\pi M H K_{eff}}{\mu R T \ln(r_o/r_i)} P^2 = \frac{\pi M H K_{\infty} P_0^2}{\mu R T \ln(r_o/r_i)} (\Pi^2 - 1) \left(1 + \frac{b}{\Pi + 1}\right) \quad (13)$$

$$K_{eff} = K_{\infty} \left[1 + \frac{b}{(\Pi + 1)}\right] = K_{\infty} \left[1 + \frac{2B}{P_0(\Pi + 1)}\right] \quad (14)$$

Here B is the Klinkenberg coefficient with pressure units and $b = 2B/P_0$ its dimensionless form referenced to $\Pi = P/P_0$. The equivalent capillary-model form is

$$G = \frac{\pi M N D^4 P_0^2}{16 \mu R T L \ln(r_o/r_i)} (\Pi^2 - 1) \left(1 + 4 K n_0^{(cap)} \frac{2 - \Sigma}{\Sigma} \frac{1}{\Pi + 1}\right) \quad (15)$$

Equations (7) and (8) yield analytical expressions for K_{∞} whose geometric parameters (N, h, D) can be estimated from independent pore-structure measurements, whereas K_{∞} and B (or b) are obtained from gas-permeability fits.

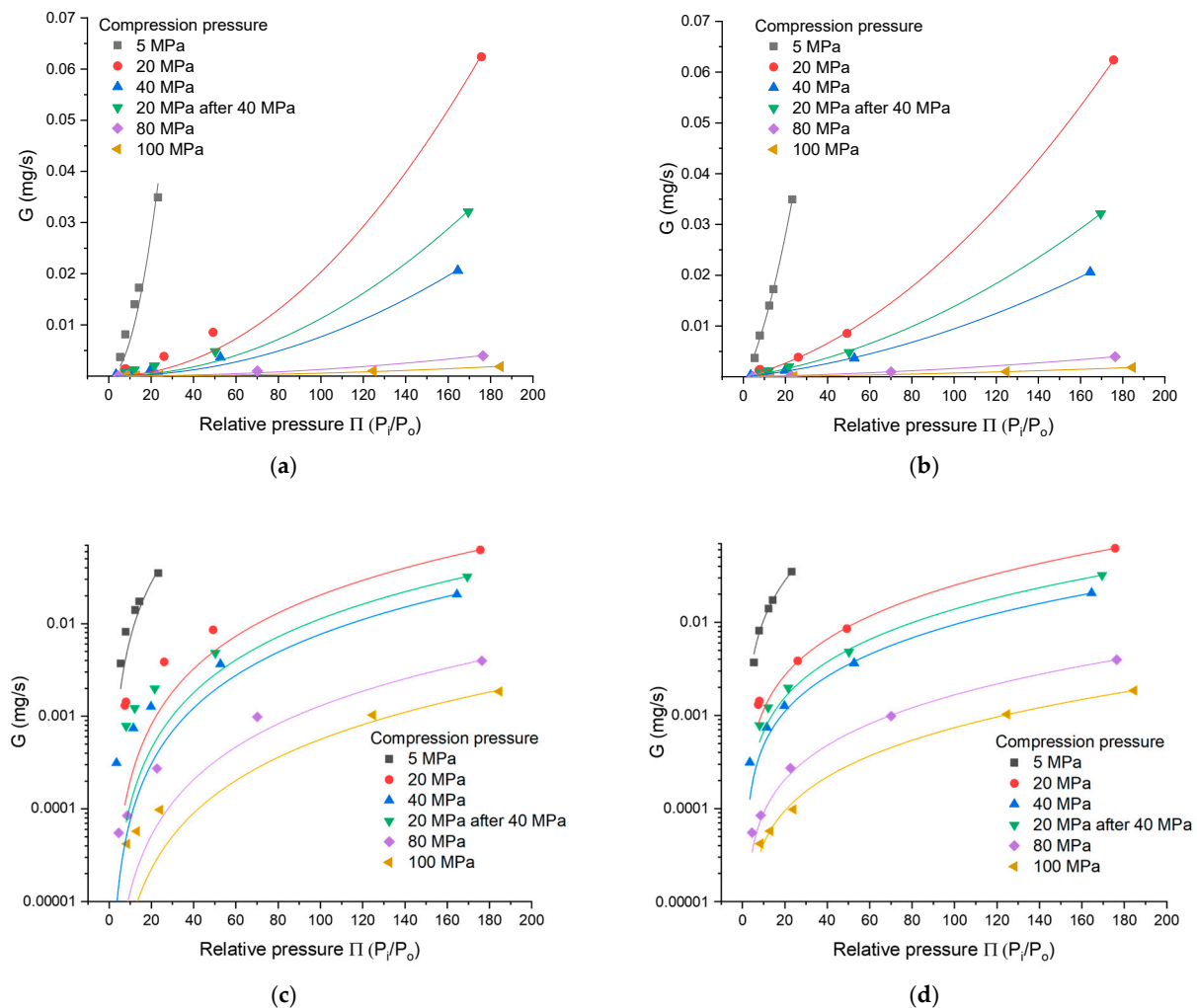


Figure 1. Helium permeability versus normalized medium pressure Π for graphite-foil gaskets. Fits of the first-order (a,c) and second-order (b,d) slot models are shown in linear (a,b) and logarithmic (c,d) representations. Markers—experimental data; lines—model fits.

Figure 1 shows the fitting results in the first-order (Figure 1a,c) and second-order (Figure 1b,d) slot models in linear and logarithmic coordinates. The first-order model fails

to reproduce the low-permeability region, which is clearly visible on the logarithmic plots, whereas the second-order model captures the dependence over the whole measured range. The fitting results are summarized in Table 3.

In the first-order model, both the apparent permeability values and the residuals are larger because the model lacks slip-flow effects. The second-order model gives a high level of agreement with the data over the whole permeability range. The fitted capillary sizes are consistent with our previous pore-size measurements by mercury porosimetry [20] and with the values reported in [17] for compression in the range of 2–14 MPa. The monotonic decrease in capillary size with compression pressure observed here differs from the nearly linear dependence reported in [17]. Figure 2 shows the dependence of capillary diameter and capillary count on compression pressure, together with linear fits. The data are of the same order of magnitude; the differences between our and literature values are most likely caused by differences in the graphite foils used. We tentatively assume that in the range 15–20 MPa, where the foil density reaches 1.4–1.5 g/cm³, the dominant compression mechanism of graphite foil changes, in agreement with the earlier observations of Ionov on the tensile strength and elastic modulus of graphite foil [32]: this corresponds to a saturation of inter-particle contacts and a transition from a plateau-like deformation regime to densification.

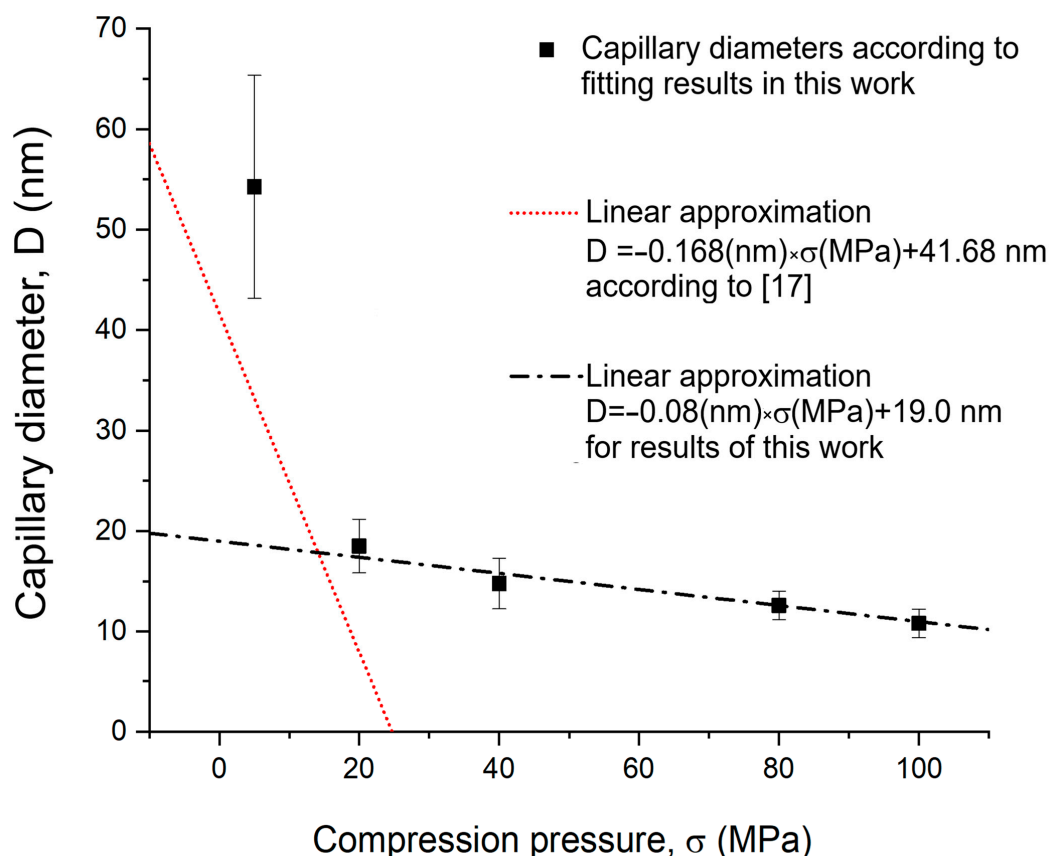


Figure 2. Capillary diameter and capillary count obtained from the second-order Darcy–Klinkenberg fit (square markers), compared with the linear dependence reported in [17] (red dotted line). Black dash-dotted line—linear fit of the capillary count versus compression pressure in the 20–100 MPa range.

Table 3. Fitted parameters of the first- and second-order permeability models for graphite foil and comparison with literature data [17]. Numbers in parentheses are the estimated standard errors in the last digit.

σ , MPa	K_{eff} , nm ² (1st Order)	R^2 (1st)	K_{∞} , nm ² (2nd, D–K)	B , MPa (2nd, D–K)	N (Slit)	$2h$, nm (Slit)	D , nm (cap)	$N_D \times 10^8$ (cap)	R^2 (2nd)	σ_{lit} , MPa [17]	D_{lit} , nm [17]	$N_{D,lit} \times 10^8$ [17]
5	60 (5)	0.916	26 (3)	0.7 (1)	693 ± 119	81 (13)	54 (11)	11 (1)	0.9985	4.13/6.21 *	35.1/31.4 *	24.6/20.4
20	4.3 (2)	0.992	3.5 (2)	1.4 (2)	1466 ± 189	28 (2)	18 (3)	70 (3)	0.99982	13.79	19.07	9.53
40	1.58 (7)	0.987	0.97 (7)	1.9 (3)	873 ± 143	22 (3)	15 (3)	52 (4)	0.99938	—	—	—
20 (after 40)	2.4 (1)	0.989	1.6 (1)	1.6 (3)	1034 ± 346	25 (8)	16 (3)	55 (4)	0.99965	—	—	—
80	0.34 (2)	0.984	0.20 (1)	2.0 (2)	271 ± 44	19 (2)	13 (2)	18.9 (9)	0.99984	—	—	—
100	0.163 (7)	0.986	0.09 (1)	2.3 (3)	186 ± 160	16 (4)	11 (1)	15.1 (9)	0.99994	—	—	—

* 4.13 MPa and 6.21 MPa are two nearest measurement points to 5 MPa from [17].

3.3. Permeability and Klinkenberg Coefficient Versus Deformation

The practically important task is to model gas permeability versus medium pressure during relaxation, since it directly mimics gasket behavior during service. Kobayashi [37] proposed and verified for asbestos-free pressed sheet gaskets of different sizes a simplified empirical dimensionless model with an exponential dependence of leakage on engineering strain ε and a power-law dependence on medium pressure P (where α , ω and H denote the inner diameter, width and height of the gasket, respectively):

$$G = 0.01354 \times H \frac{\alpha}{\omega} e^{-57.8\varepsilon} P^{1.35} \quad (16)$$

It is worth checking whether an analogous approach is applicable to the parameters of the laminar–molecular flow (LMF) model. Slot and capillary representations are preferred for comparison with porosimetry data; the Darcy–Klinkenberg representation is convenient for dimensionless modeling. Figure 3 shows the dependence of the intrinsic permeability K_{∞} and the Klinkenberg coefficient B on the engineering compressive strain $\varepsilon = 1 - H/H_0$. Both quantities are well described by exponential functions, which supports the use of exponential dependences on strain for the parameters of the second-order permeability model in graphite foil.

The exponential dependence of K_{∞} on strain is physically consistent with the known deformation and pore-structure evolution in graphite gaskets and packings under compression [18,26]. Mercury porosimetry and small-angle neutron scattering studies [5,6,19,20] have shown that the dominant pore size decreases from ~100 nm to ~4 nm as compression increases from ambient to 100 MPa, with the decrease being approximately exponential in the intermediate range. Since for both slit and capillary geometries K_{∞} scales as a power of the characteristic pore size (h^3 or D^4 , respectively), an exponential dependence of K_{∞} on strain follows naturally if the characteristic pore dimension is itself exponentially related to strain—which is consistent with the Cam Clay-type logarithmic compression framework introduced in Section 3.4. The increase in B with decreasing compression (i.e., increasing strain during unloading) reflects the increase in the effective Knudsen number as larger pores re-open upon load removal. The present data do not permit separation of the contributions from changes in pore count N and pore size, which would require complementary in situ porosimetry; this would be a subject for further study.

The exponential dependences obtained for K_{∞} and B allow Equation (13) to be transformed into a form similar to the empirical Kobayashi equation [37]. Assuming

$$K_{\infty} = C_K e^{Z_K \times (\varepsilon)} \quad (17)$$

$$P_0^2 (\Pi^2 - 1) \left(1 + \frac{b}{\Pi + 1}\right) \sim P^x \quad (18)$$

one obtains an expression with a power-law dependence on the medium pressure and an exponential dependence on the strain:

$$G \sim Ae^{Z_{K \times (\varepsilon)} p^x} \quad (19)$$

where A is a coefficient that absorbs the geometrical factors of the gasket and the properties of helium at normal temperature and pressure, and x is a dimensionless exponent describing the dependence on the medium pressure ($1 \leq x \leq 2$).

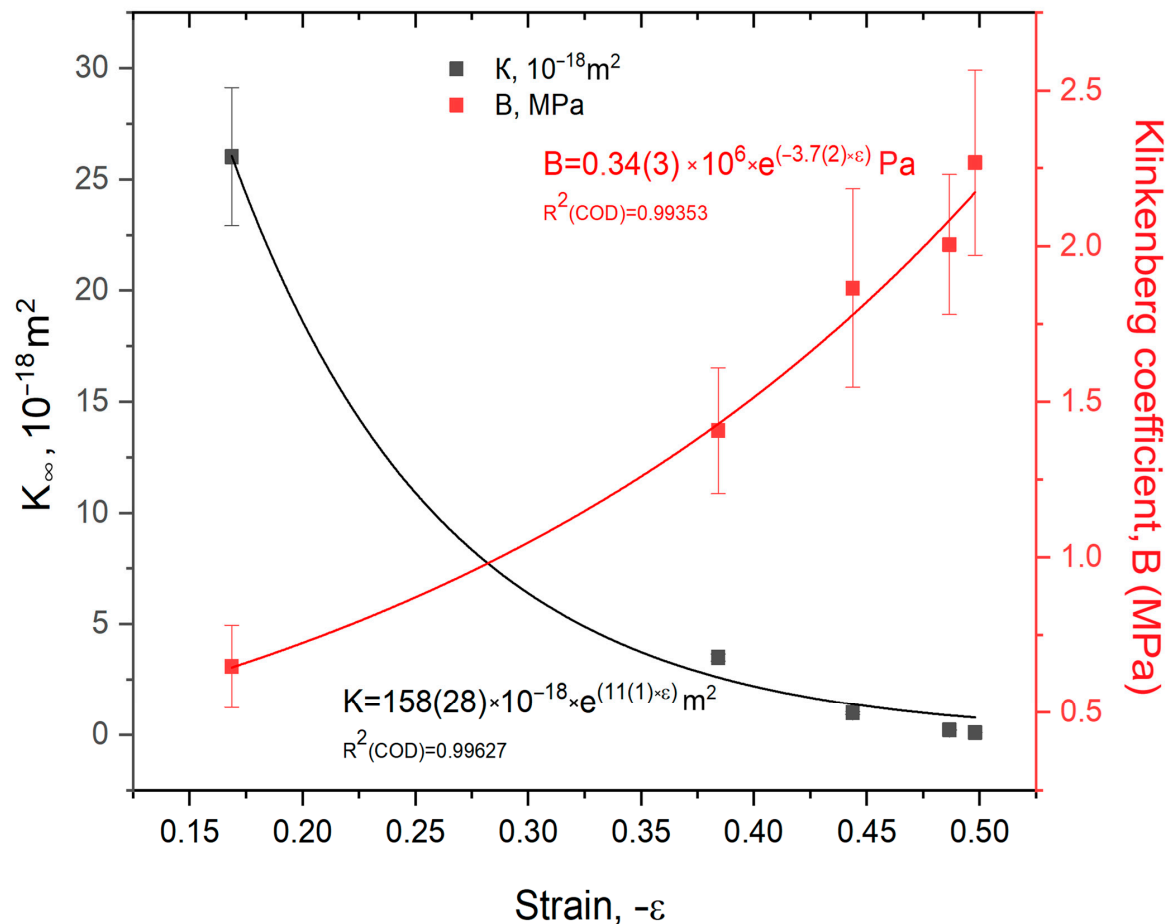


Figure 3. Intrinsic permeability K_{∞} and Klinkenberg coefficient B as functions of the compressive strain ε from the fits of the second-order model (markers), approximated by exponential functions (lines).

3.4. Compression Pressure Versus Deformation

For practical application, the dependence of permeability on the compression pressure is the most important parameter, since the compression pressure is the target parameter in flange-joint analysis and is also the basic measured variable in EN 13555 tests. Figure 4 shows loading and unloading curves for graphite-foil specimens in logarithmic coordinates. The dependence of compression pressure on strain during unloading is well described by exponential functions. During loading, an inflection is observed at compression pressures of 20–30 MPa (strain ≈ 0.4), in agreement with the conclusion of Ionov [32].

The following Cam Clay-inspired compression dependence was used to describe the compression-pressure–strain curves:

$$\bar{\sigma} = e^{Y \times (\varepsilon - \varepsilon_{\sigma})} - 1 = Ae^{Y(\varepsilon)} - 1 \quad (20)$$

where $\bar{\sigma} = \sigma/\sigma_{\text{ref}}$ is the dimensionless compression pressure, ε is the engineering compressive strain, and ε_{σ} is the residual (plastic) strain after complete unloading from the corresponding maximum compression pressure σ_0 , $A = e^{-Y\varepsilon_{\sigma}}$. σ_{ref} was set to 1 MPa for convenience so that $\bar{\sigma}$ numerically coincides with σ expressed in MPa. Differentiating Equation (20) gives the effective tangent modulus:

$$E(\varepsilon) = \frac{d\sigma}{d\varepsilon} = AY e^{Y(\varepsilon)} = \sigma_{ref} Y [\bar{\sigma}(\varepsilon) + 1] \quad (21)$$

which is reported for the point of unloading initiation in Table 4. Equation (20) does not adequately describe the loading branch at low compression pressures (Figure 5a); splitting the curve into two ranges ($\varepsilon < 0.4$ and $\varepsilon > 0.4$) allows the loading dependence to be fitted correctly (Figure 5b). For unloading curves, Equation (20) gives a good fit over the entire measured range (Figure 5c). The effective tangent moduli obtained from Equation (21) fall between the moduli measured by the EN 13555 and DIN 28090-2 [38] standards, in which the unloading modulus is determined by a linear fit of the upper two-thirds of the unloading branch with the strain referenced to either the compressed or the original specimen thickness. The values from Equation (21) are by construction effective tangent moduli. The loading modulus is not reported because of the predominantly elasto-plastic deformation under loading. The compression and unloading branches of graphite foil can be interpreted in a Cam Clay-type logarithmic stress–strain framework. Taking the logarithm of (20) gives $\varepsilon = \varepsilon_\sigma + Y^{-1} \ln(\bar{\sigma} + 1)$, which is formally analogous to the $e\text{-}\ln(p)$ compression lines used in Cam Clay models. In this analogy, Y^{-1} is an effective compressibility index, while ε_σ defines the branch-specific structural state of the compressed graphite foil. A more complete constitutive description of graphite foil would incorporate plastic strains through a yield surface and a hardening law—analogous to the modified Cam Clay model in soil mechanics, or through continuum damage mechanics if pore collapse is treated as a damage process. Such a framework would naturally capture the divergence of the loading and unloading branches and provide a physically motivated basis for the Y and ε_σ parameters. Development of a constitutive model for exfoliated graphite under cyclic loading is a promising direction for further study.

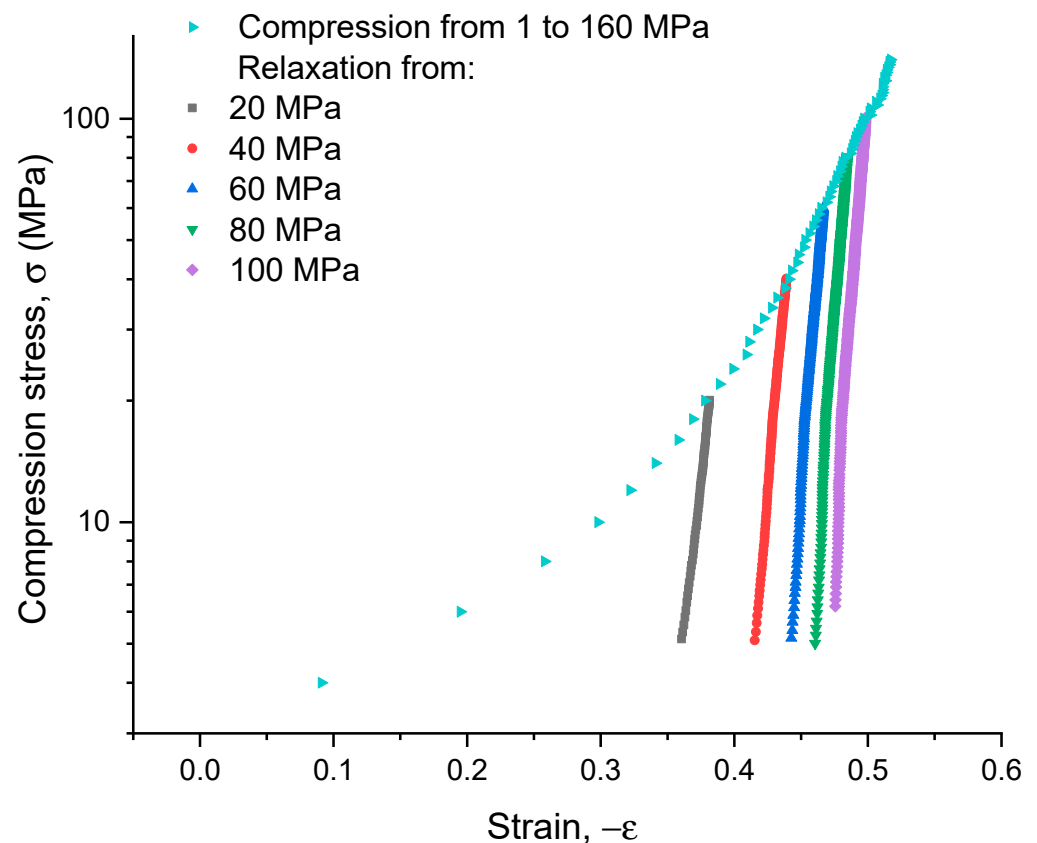


Figure 4. Experimental dependence of compression pressure on strain during loading and unloading from prescribed compression pressures, plotted in logarithmic coordinates.

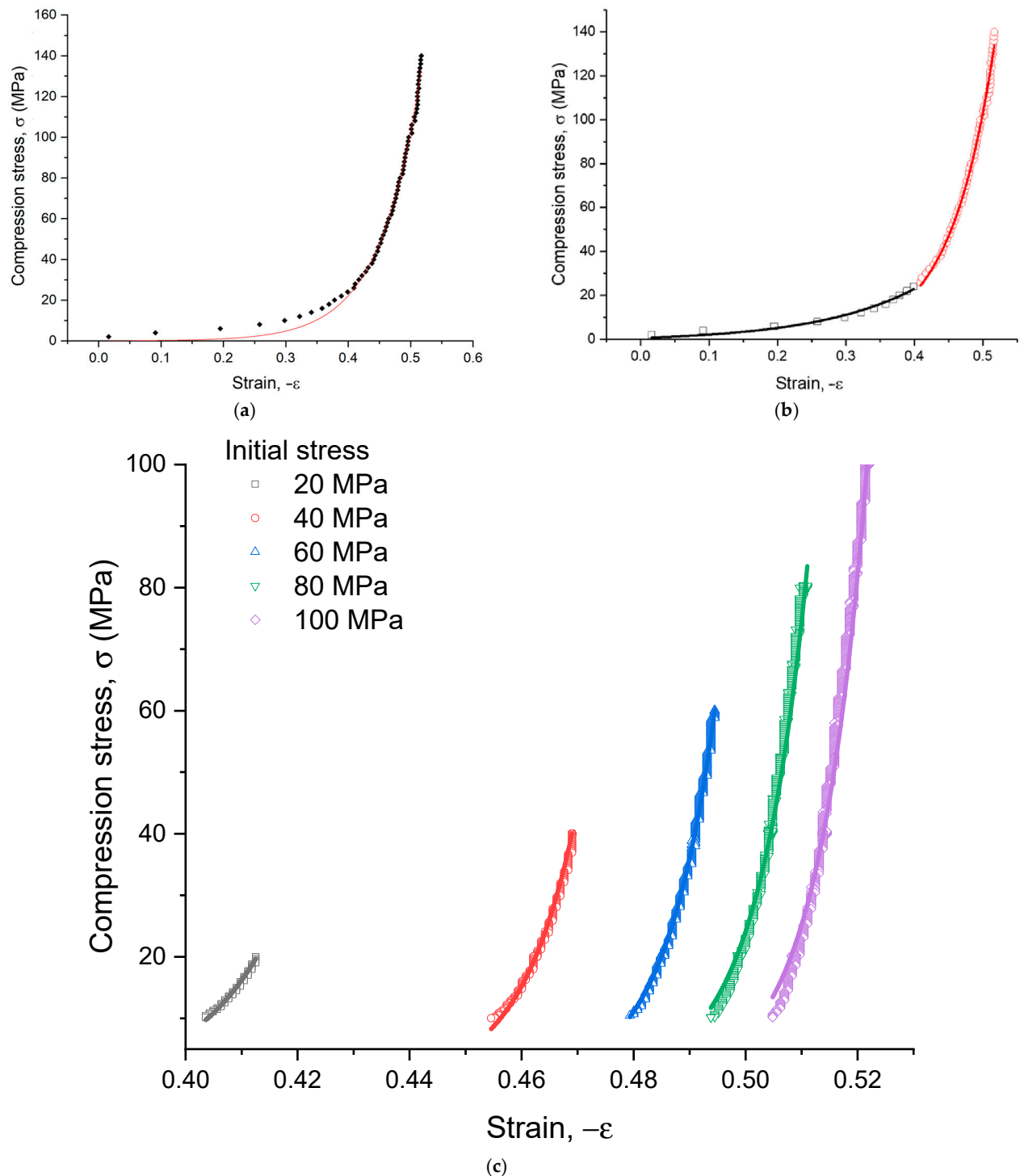


Figure 5. Fits of the compression-pressure-strain dependence using Equation (20): (a) loading curve fitted as a single function; (b) loading curve split into two ranges ($\epsilon < 0.4$ (black) and $\epsilon > 0.4$ (red)); (c) unloading curves from different maximum compression pressures. Markers — experimental data; lines — fits.

Table 4. Parameters of Equation (20) for the compression-pressure-strain dependence of graphite foil.

Parameter	Loading, Single Fit	Loading, $\epsilon < 0.4$	Loading, $\epsilon > 0.4$	Unloading from 20 MPa	From 40 MPa	From 60 MPa	From 80 MPa	From 100 MPa
Y	15.3 (2)	6.8 (5)	15.4 (2)	64.2 (6)	79.9 (4)	87.7 (4)	93.0 (4)	95.3 (3)
$\epsilon\sigma$	0.05 (1)	−0.06 (3)	0.199 (3)	0.334 (4)	0.392 (2)	0.420 (2)	0.437 (2)	0.450 (2)
$E(\epsilon) = \sigma_{ref} Y(\bar{\sigma} + 1)$ at start of unloading MPa				1348	3276	5350	7533	9625

E per EN 13555, MPa				777	2864	4700	5736	6234
E per DIN 28090-2, MPa				1286	5138	8869	11145	12365
R ²	0.99452	0.97634	0.99399	0.99614	0.99713	0.99704	0.99700	0.99773

3.5. Permeability Versus Compression Pressure During Relaxation

Representing the exponential dependences of K_∞ and B on strain as

$$K_\infty = C_K e^{Z_K(\epsilon)} \quad B = C_B e^{Z_B(\epsilon)} \quad (22)$$

and using Equation (20) to eliminate ϵ , the parameters can be re-expressed as power laws of the dimensionless compression pressure $\bar{\sigma} = \sigma/\sigma_{\text{ref}}$:

$$K_\infty = C_{K1}(\bar{\sigma} + 1)^{W_K} \quad B = C_{B1}(\bar{\sigma} + 1)^{W_B} \quad (23)$$

where

$$\begin{aligned} C_{K1} &= C_K e^{Z_K(\epsilon_\sigma)} & C_{B1} &= C_B e^{Z_B(\epsilon_\sigma)} \\ W_K &= Z_K/Y & W_B &= Z_B/Y \end{aligned} \quad (24)$$

Power-law fitting of $K_\infty(\bar{\sigma})$ and $B(\bar{\sigma})$ (Figure 6) yields good agreement; the fitted parameters are summarized in Table 5.

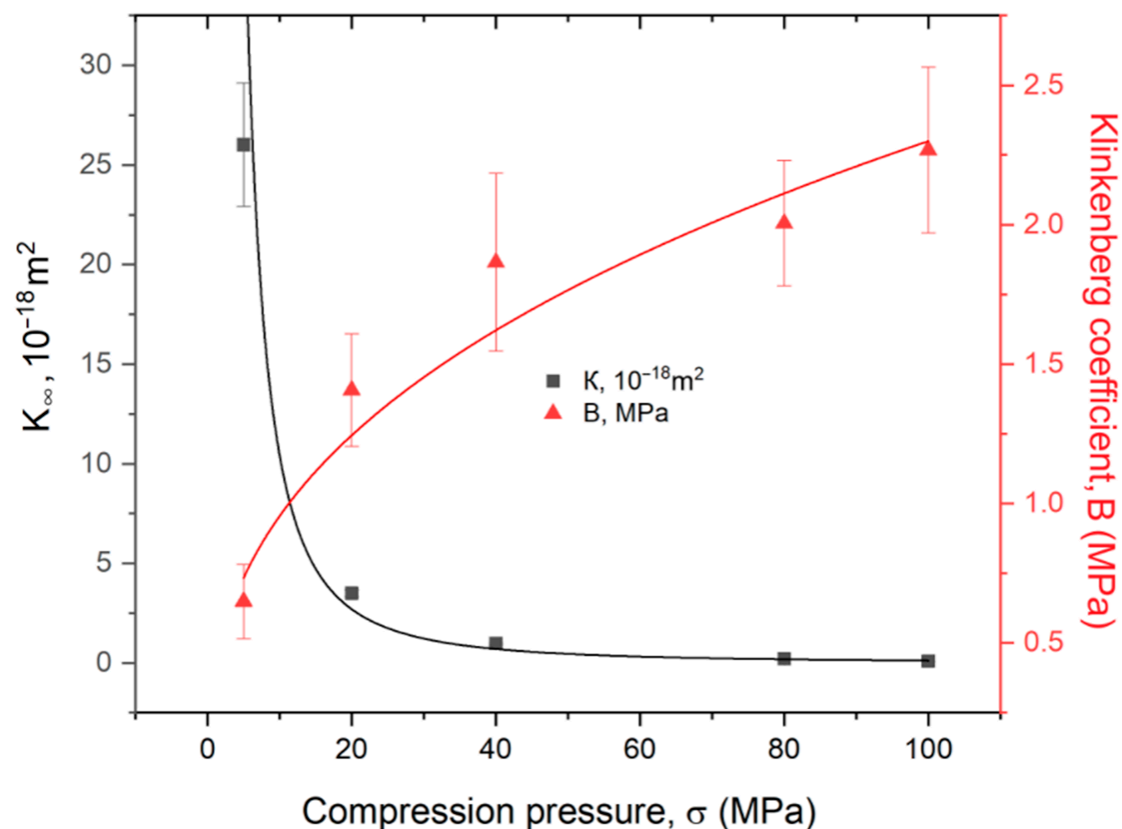


Figure 6. Intrinsic permeability K_∞ and Klinkenberg coefficient B as functions of the compression pressure, fitted by power-law functions (Equation (23)). Markers—experimental data; lines—fits.

Table 5. Parameters of the exponential and power-law dependences of K_∞ and B (Equations (22) and (23)).

Quantity	K_∞	B
Exponential form (Equation (22))	$C_K = 158 (28) \times 10^{-18} \text{ m}^2$ $Z_K = 11 (1)$	$C_B = 0.34 \times 10^6 \text{ Pa}$ $Z_B = -3.7 (2)$
Power-law form (Equation (23))	$C_{K1} = 488 (32) \times 10^{-18} \text{ m}^2$ $W_K = -1.63 (3)$	$C_{B1} = 0.36 (8) \times 10^6 \text{ Pa}$ $W_B = 0.40 (5)$

Combining Equations (13) and (23) yields the leakage rate as a function of the dimensionless compression pressure and the relative medium pressure:

$$G(\bar{\sigma}, \Pi) = \frac{\pi M H C_{K1} (\bar{\sigma} + 1)^{W_K} P_0^2}{\mu R T \ln(\bar{r}_o / \bar{r}_i)} (\Pi^2 - 1) \left(1 + \frac{2 C_{B1} (\bar{\sigma} + 1)^{W_B}}{P_0 (\Pi + 1)} \right) \quad (25)$$

with

$$C_{G1} = \frac{\pi M H C_{K1} P_0^2}{\mu R T \ln(\bar{r}_o / \bar{r}_i)} \quad (26)$$

Introducing the positive parameter $\gamma = -W_K/W_B$ and using Equations (22) and (23), the ratio $W_K = -\gamma W_B$ is obtained. The parameter $\gamma = -W_K/W_B$ can be estimated independently from the fitted exponential coefficients: $W_K = -1.63$ (3) and $W_B = 0.40$ (5) (Table 5) give $\gamma \approx 4.0 \pm 0.3$. This value lies between the slit-model prediction ($\gamma_{\text{slit}} = 3$, since $K_\infty \sim h^3$ and $b \sim 1/h$) and the capillary-model prediction ($\gamma_{\text{cap}} = 4$, since $K_\infty \sim D^4$ and $b \sim 1/D$) and is closer to the capillary model. The capillary model approach is consistent with the observed channel dimensions and their comparison with mercury porosimetry data [20]. Adopting $\gamma = 3$ instead of 4 would change the exponents in Equation (27) by less than 10% (since W_B itself varies by $\sim 30\%$ across unloading branches, Table 6), confirming that the simplified analysis is robust to this choice within the experimental uncertainty. Accordingly, the obtained value of γ is consistent with a capillary-type flow through narrow channels; however, the present data alone do not yet allow the contributions of varying channel number and channel size to be disentangled.

Assuming $\gamma = 4$ for a simplified analysis of permeability versus compression pressure at constant medium pressure $\Pi = \text{const}$, Equation (25) becomes:

$$G(\bar{\sigma}) = C'_{G1} (\bar{\sigma} + 1)^{-4W_B} + C'_{G2} (\bar{\sigma} + 1)^{-3W_B} \quad (27)$$

With

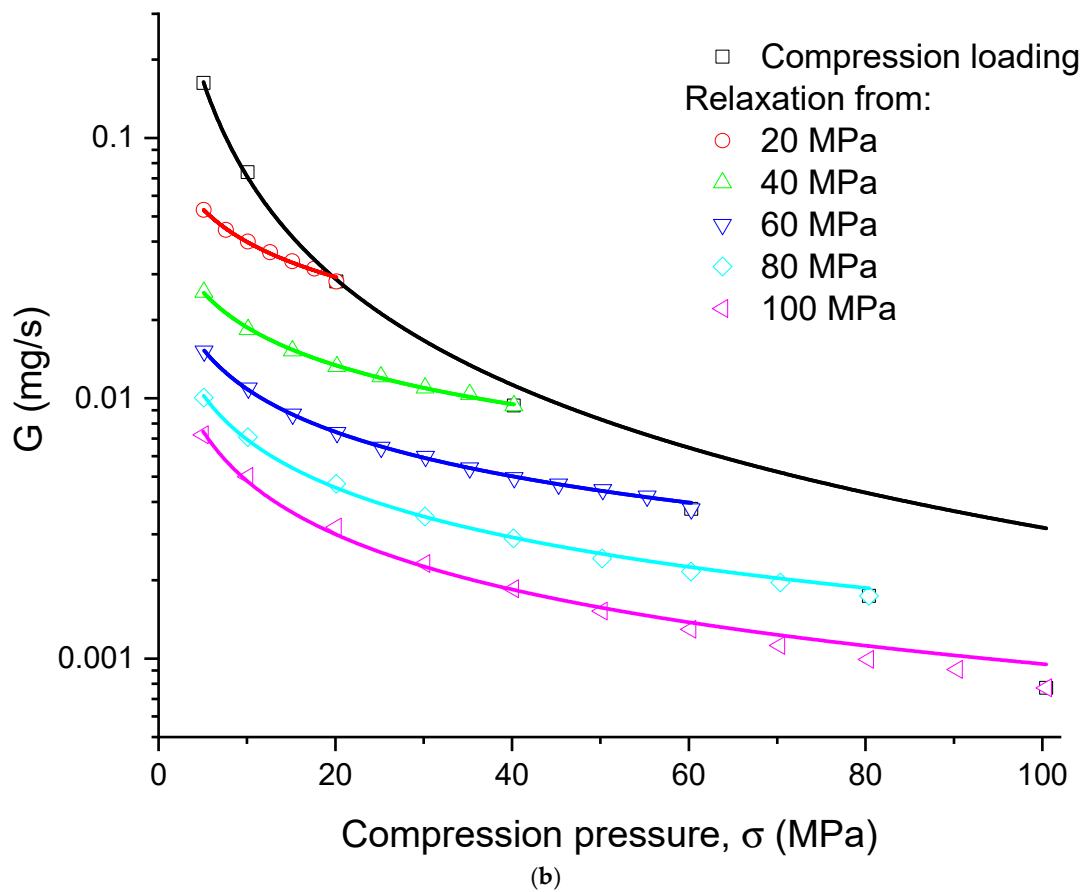
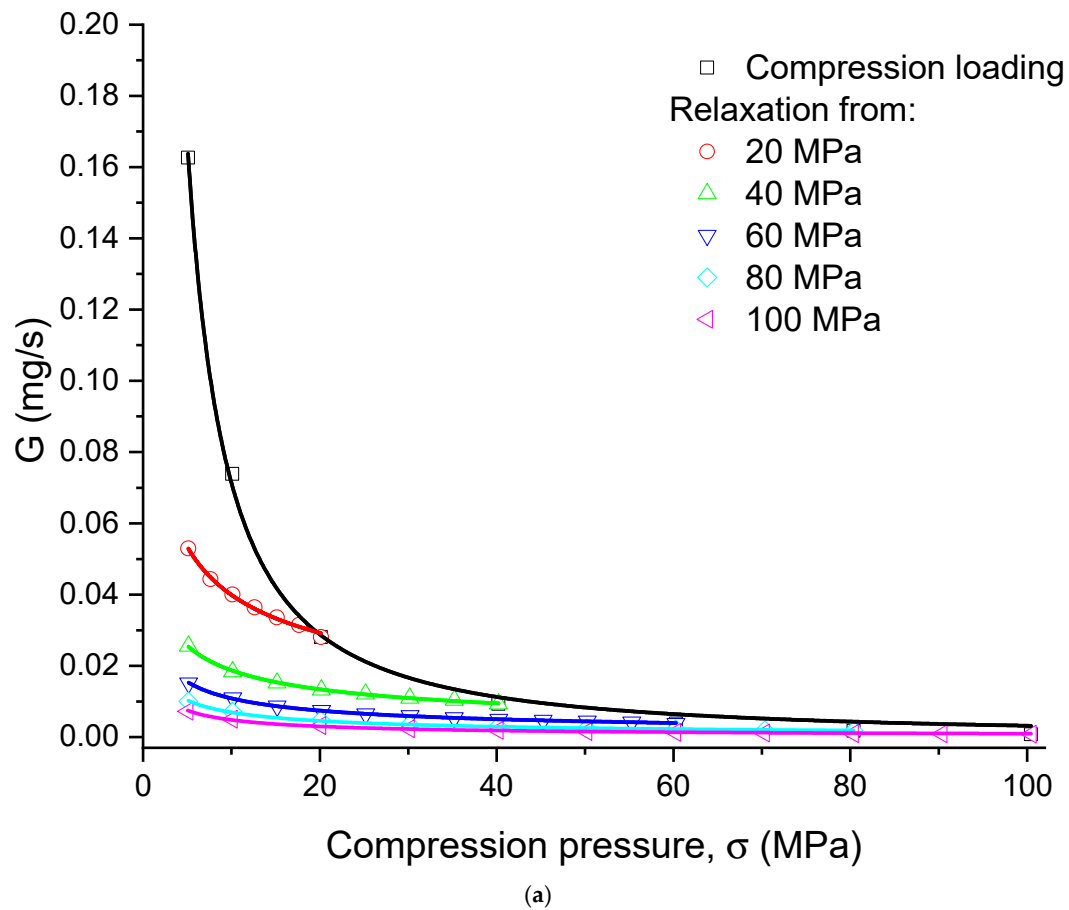
$$C'_{G1} = \frac{\pi M H C_{K1} P_0^2}{\mu R T \ln(\bar{r}_o / \bar{r}_i)} (\Pi^2 - 1); \quad C'_{G2} = \frac{2 C'_{G1} C_{B1}}{P_0 (\Pi + 1)} \quad (28)$$

The corresponding first-order equation:

$$G(\sigma) = C'_{G1} (\bar{\sigma} + 1)^{-W_K} = C'_{G1} (\bar{\sigma} + 1)^{-4W_B} \quad (29)$$

Figure 7 shows the fits of the second-order model in linear (Figure 7a) and logarithmic coordinates (Figure 7b). The unloading branches are well described by both the first- and the second-order forms, the second order being preferable at low permeability. Positivity constraints were imposed on K_∞ during fitting and the fitting curves and values for first and second order models were practically equivalent (Table 6).

The loading branches at low permeability are not described as well when represented in logarithmic coordinates (Figure 7b), which is consistent with the earlier observation that plastic deformation modifies the channel structure irreversibly. During relaxation, no further plastic deformation occurs and the pore-structure response is reversible. We previously applied partial fitting of the loading stress via strain (Figure 5b) for compressive strain $-\epsilon > 0.4$ and compressive pressure more than 20 MPa. This approach was adopted in line with Ionov's [32] observation of change for mechanical properties. Exclusion of the initial region of loading with $\sigma < 20$ MPa leads to a more appropriate fit even in the low permeability region when represented in logarithmic coordinates (Figure 7c) and could be used for engineering practice. The fitted parameters are listed in Table 6. The first- and second-order descriptions are practically equivalent up to a factor in the power-law exponent.



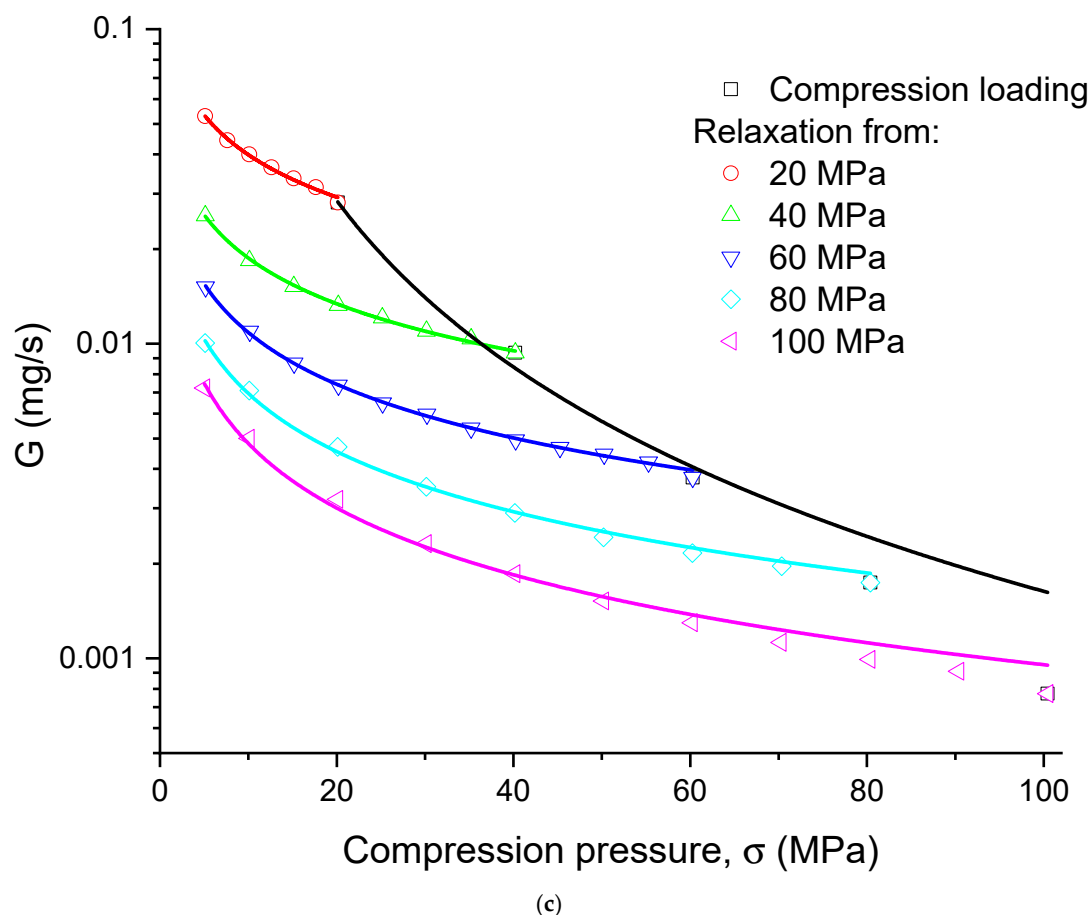


Figure 7. Helium permeability as a function of the dimensionless compression pressure σ^- in the second-order model: (a) linear scale; (b) logarithmic scale; (c) logarithmic scale with loading branch fitted only for $\sigma > 20$ MPa. Markers—experimental data; lines—fits.

Table 6. Parameters of Equations (27) and (29) for permeability versus compression pressure *.

Model	Parameter	Loading	Loading in the Region of 20–100 MPa (Figure 7c)	Unloading from 20 MPa	From 40 MPa	From 60 MPa	From 80 MPa	From 100 MPa
1st order	C_{G1}	2.1 (2)	7.3 (1)	0.126 (4)	0.065 (1)	0.044 (1)	0.034 (1)	0.028 (1)
	$W_B = -W_K/4$	0.35 (1)	0.45 (1)	0.120 (3)	0.130 (6)	0.147 (1)	0.164 (3)	0.182 (5)
	R^2	0.99835	0.9999	0.99527	0.99899	0.99919	0.99797	0.99497
2nd order	C_{G1}	2.1 (4)	7.3 (1)	0	0	0	0	0
	W_B	0.35 (1)	0.45 (1)	0.15 (19)	0.15 (4)	0.20 (3)	0.22 (6)	0.24 (8)
	C_{G2}	0	0	0.12 (6)	0.03 (6)	0.04 (3)	0.03 (5)	0.027 (4)
	R^2	0.99835	0.9999	0.99527	0.99910	0.99919	0.99795	0.99487

* Numbers in parentheses are estimated standard errors in the last digit(s), obtained from the non-linear least-squares fit (Levenberg–Marquardt). Large relative errors for the 2nd-order W_B parameter in the unloading series reflect the low sensitivity of the $G(\sigma)$ data to B at high compression, as discussed in the text (Section 3.5).

A robust evaluation of the Klinkenberg coefficient B from the permeability versus compression-pressure data at fixed medium pressure was not achieved because the dependence is not sensitive enough; B is therefore better obtained from the $G(P)$ data at fixed compression pressure (Equation (13)). With this caveat, the first-order expression provides comparable accuracy to the second-order one. Consequently, the 2nd-order C_{G1} values converge to zero for the unloading branches (Table 6), and the corresponding W_B standard errors are large; these results should not be interpreted as a failure of the model

but rather as a reflection of the limited sensitivity of the compression-pressure dependence to slip flow at high compression levels

The dependences of the parameters C_{G1} and W_B on compression pressure are monotonic and can be described by a power-law and a linear function, respectively (Figure 8).

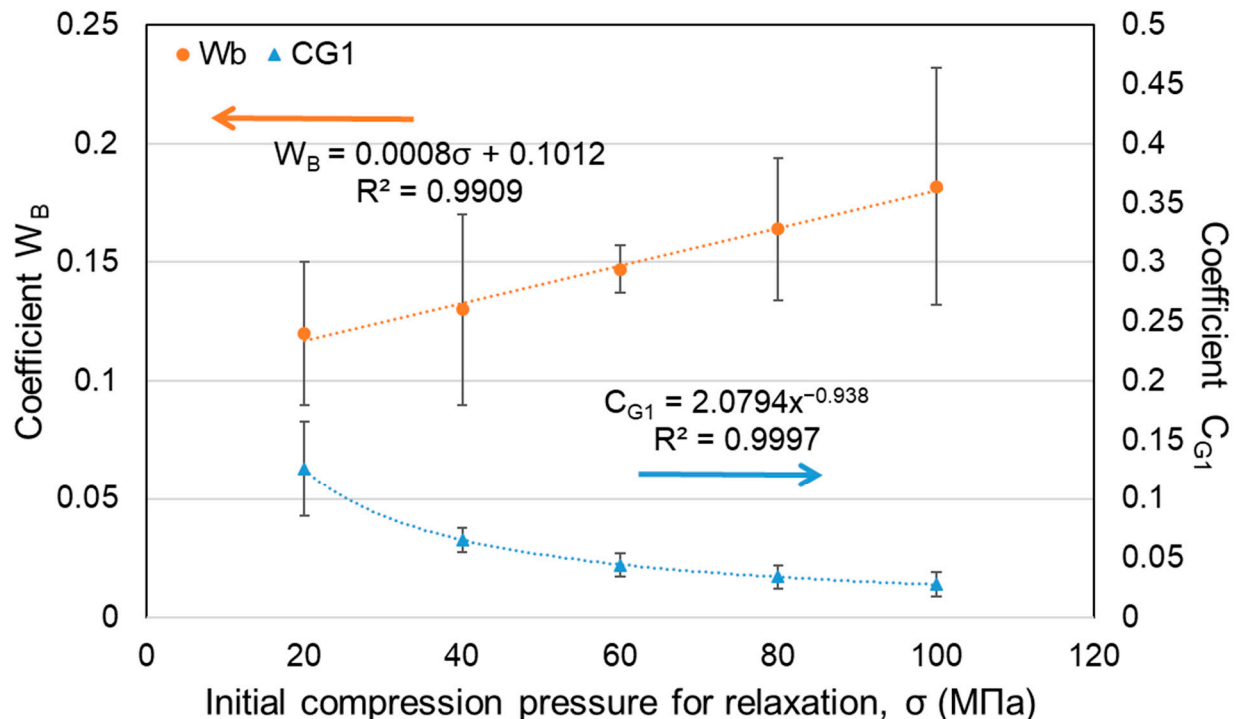


Figure 8. Coefficients C_{G1} (blue color) and W_B (orange color) as functions of the maximum compression pressure σ in the second-order model. Markers—experimental data; lines—fits.

The data in Table 6 can be used to compute permeability coefficients with Equation (25) for arbitrary gasket geometries and medium pressures using the following relations:

$$C_{G1} = \frac{\pi M H C_{K1} P_0^2}{\mu R T \ln(r_o/r_i)} \quad (30)$$

$$K = C_{K1}(\bar{\sigma} + 1)^{W_K} \quad (31)$$

$$\frac{C'_{G1}}{C_{K1}} = 4.175 \times 10^{12} \times H / \ln(r_o/r_i) \frac{kg}{(m^2 s)} \quad (32)$$

For a gasket of thickness 1 mm with outer and inner diameters of 88 mm and 49 mm, this ratio is approximately $7.124 \times 10^9 \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$, i.e.,

$$K = C_{K1}(\bar{\sigma} + 1)^{W_K} = 1.4037 \times 10^{-10} \frac{(m^2 s)}{kg} C_{G1}(\bar{\sigma} + 1)^{W_K} \quad (33)$$

Combining this representation with the Darcy–Klinkenberg model produces a universal dimensionless mathematical model of the dependence of permeability of graphite foil on compression and medium pressures. Equation (25), together with its first-order simplification, provides a satisfactory description of permeability during stress relaxation, allowing the gasket response in real service to be modeled numerically.

The proposed correlation between leakage-channel size and macroscopic compressive strain is supported by the monotonic decrease in fitted channel dimensions with compression pressure and by previously reported pore-structure changes in compressed ex-

foliated graphite. However, graphite foil is anisotropic, and the evolution of in-plane leakage channels may differ from through-thickness pore closure. The proposed relation should therefore be regarded as an effective phenomenological description within the calibrated pressure range rather than a direct microstructural law. The model is valid for Knudsen numbers below unity and within the compressive-strength limit of the foil (about 100–150 MPa for 1.5–2 mm thick specimens). Creep, viscoplasticity and strain-rate effects introduce additional uncertainties in the mechanical part. Permeability is in general a tensor; the present model assumes a dominant in-plane component and is therefore a two-dimensional approximation. A separate investigation of the tensor components is required for fully three-dimensional flange-joint modeling.

A qualitative comparison with previously published models is possible on the basis of graphical data reported in the respective papers. Two models in the literature address the same combination of leakage versus compressive and medium pressures: the empirical model of Kobayashi et al. [37] (Equation (16)) and the capillary-based prediction of Gu et al. [25]. The model of Gu et al. [25] provides a satisfactory description of leakage as a function of medium pressure at moderate compression levels (laminar–molecular flow regime), but its predictions for leakage as a function of compressive stress show appreciable deviations from experimental data, as acknowledged by the authors themselves. The model of Kobayashi et al. [37] describes leakage as a function of compressive stress during unloading more accurately than Gu et al. [25]; however, as is evident from Figure 11 of ref. [37], significant scatter remains, which may be attributed to the use of fixed universal empirical coefficients that do not reflect the specific mechanical and microstructural properties of the gasket material. Neither paper reports quantitative goodness-of-fit statistics, so a formal quantitative benchmarking is not feasible from the published data; only a graphical comparison is possible. In contrast, the proposed model (Equation (25)) was fitted individually to the experimental dataset of the present study, yielding coefficients of determination $R^2 \geq 0.9985$ across all compression levels and medium pressures, which represents a substantially improved description of the compression–permeability relationship under stress-relaxation conditions.

4. Conclusions

A semi-empirical model based on the Darcy–Klinkenberg formulation has been developed and validated for predicting gas permeability of graphite foil gaskets as a function of compression pressure and medium pressure during stress relaxation. The key findings are:

1. The second-order Darcy–Klinkenberg model describes the dependence of helium leakage on medium pressure significantly better than the first-order Darcy approximation across all tested compression levels ($R^2 \geq 0.9985$ vs. 0.916–0.992), particularly in the low-leakage regime where slip flow is dominant.
2. The intrinsic permeability K_∞ and the Klinkenberg coefficient B follow exponential functions of compressive strain ε ($R^2 \approx 0.996$ and 0.994, respectively) and power-law functions of dimensionless compression pressure σ ($R^2 > 0.98$ for both). These dependences are physically consistent with the known pore-structure evolution in compressed exfoliated graphite.
3. Combining the Darcy–Klinkenberg model with the power-law compression-pressure dependences yields Equation (25), which predicts gas leakage during unloading with $R^2 > 0.995$ across relaxation branches from 20 to 100 MPa. Model coefficients can be determined from standard EN 13555 leakage tests performed at various medium pressures. The same equation describes permeability during loading, with reduced accuracy at high compression pressures owing to irreversible plastic pore-structure modification.

4. The model's applicability is bounded by the compressive strength of the foil (~100–150 MPa for 1.5–2 mm specimens), Knudsen numbers below unity, and the calibrated compression range. Further limitations and future directions are discussed in Section 3.5.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/pr14132105/s1>, Figure S1: XRD pattern of intercalated graphite nitrate. Figure S2: Photographs of (a) a representative graphite foil sheet as prepared by uniaxial pressing and (b) an annular test specimen. Tables S1–S3: Extended versions of Tables 3, 4 and 6 of main manuscript with additional goodness of fit parameters—Adjusted R^2 , and RMSE.

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Data Availability Statement: Data available upon request.

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Conflicts of Interest: The author declares no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

GF	Graphite foil
EG	Exfoliated (thermally expanded) graphite
LMF	Laminar–molecular flow model
LMA	Levenberg–Marquardt algorithm
D–K	Darcy–Klinkenberg formulation
B	Klinkenberg coefficient (pressure units), Pa
$b = 2B/P_0$	Dimensionless Klinkenberg coefficient referenced to $\Pi = P/P_0$
C_K, C_B, C_{K1}, C_{B1}	Pre-exponential and pre-power-law coefficients in Equations (22) and (23)
D	Capillary diameter, nm
E	Elastic (tangent) modulus, MPa
E_c	Compressibility of the foil, %
E_r	Recovery of the foil, %
G	Helium leakage rate through the gasket (reported here in $\text{mg}\cdot\text{s}^{-1}$)
h	Slit half-height, nm
H	Gasket thickness in the compressed state, mm
H_0	Gasket thickness in the initial state, mm
Kn	Knudsen number
Kn_0	Knudsen number at normal conditions
K_{eff}	Apparent (effective) gas permeability, m^2
K_∞	Intrinsic (Klinkenberg-extrapolated) gas permeability at $\text{Kn} \rightarrow 0$, m^2
L	Capillary length (inner-to-outer radius), mm
M	Molar mass of helium, $4.003 \times 10^{-3} \text{ kg}\cdot\text{mol}^{-1}$
N	Number of slits—leakage channels in the slot model
N_D	Number of cylindrical capillaries—leakage channels in the capillary model
P	Helium pressure inside the gasket (test medium pressure), Pa
P_0	Ambient (atmospheric) pressure, 0.101325 MPa
$\Pi = P/P_0$	Dimensionless medium pressure
r_i, r_o	Inner and outer radii of the gasket, mm
R	Universal gas constant, $8.314 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$

S	Specimen cross-section, mm ²
T	Temperature, K; tests at 298 K
$W_K = Z_K/Y$	Dimensionless exponent of the σ -dependence of K_∞
$W_B = Z_B/Y$	Dimensionless exponent of the σ -dependence of B
Y	Dimensionless coefficient in Equation (20)
Z_K, Z_B	Exponential coefficients in Equation (22)
$\gamma = -W_K/W_B$	Positive ratio characterizing the relative pressure-dependences of K_∞ and B
$\varepsilon = 1 - H/H_0$	Engineering compressive strain of the gasket
ε_σ	Residual (plastic) strain after unloading from the maximum compression pressure σ_0
λ_0	Helium mean free path at normal conditions, 1.89×10^{-7} m
μ	Helium dynamic viscosity at normal conditions, 1.946×10^{-5} Pa·s
Σ	Helium–surface accommodation coefficient (taken as 1 for graphite)
σ	Compression pressure on the gasket, Pa or MPa
$\sigma^* = 1$ MPa	Reference pressure used for normalization
$\bar{\sigma} = \sigma/\sigma^*$	Dimensionless compression pressure
ρ	Specimen density, g/cm ³

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