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► To cite this version:

Maximiliano Melnichuk, Quentin Gardavaud, Frédéric Thiébaud, Dominique Perreux. Temperature effect in cavitation risk assessments of polymers for hydrogen systems. *International Journal of Hydrogen Energy*, 2020, 45, pp.23020 - 23026. <10.1016/j.ijhydene.2020.05.224>. <hal-03492061>

HAL Id: hal-03492061

<https://hal.science/hal-03492061v1>

Submitted on 22 Aug 2022

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Temperature effect in cavitation risk assessments of polymers for hydrogen systems

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Abstract

Hydrogen storage at high pressure is currently attained by the use of different materials, such as elastomers in sealing joints, thermoplastics and thermosetting polymers in high-pressure containers, and metallic tube connections. Hydrogen containers type IV use a thermoplastic polymer for hydrogen tightness and composite materials for mechanical resistance, usually made with thermosetting resins and carbon or glass fibre. International standards impose a wide range of operative temperatures for such containers, from -40°C to 85°C.

Once saturated with hydrogen at high pressure, a fast depressurisation process can create stress in the polymeric materials, causing its degradation by the formation of cavities. In a previous work, we were able to make a generalization of cavitation risk by the use of non-dimensional parameters, based on a simplified mechanical failure model. We observed that for the model, material's hydrogen diffusivity and yield strength are of utmost importance. In present work, we analyse the effect of temperature on these two properties, as they have an inverse evolution with temperature. Results confirm the pertinence of considering temperature in the whole application range of technology under analyse.

Key words:

Hydrogen storage; Explosive decompression failure; Cavitation; Non-dimensional approach; Temperature influence

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1. Introduction

Under the frame of sustainable development, hydrogen technologies may play a major role as an energy vector. In spite of some clear advantages when compared to fossil fuels, such as three times more specific energy and cleaner combustion products, hydrogen storage remains as a difficulty. Given its low density, gaseous storage usually implies the need of using high-pressure containers. A cost-effective solution is the use of composite materials, to build low-weight containers with high mechanical performance [1]. For instance, containers type IV are made with glass or carbon fibres and resin matrix, and an internal polymer liner for hydrogen tightness. International standards impose a wide range of operating temperatures for such containers, from -40°C to 85°C [2], which in turn has a remarkable influence on its constitutive polymers.

Concerning high-pressure hydrogen storage, a topic of interest is the influence of dissolved hydrogen in polymer materials, especially during depressurisation. In the case of sealing o-rings made with elastomers, there are reports of defaults formation in the form of cavities, so as fracture or blister formation during depressurisation [3-5]. This phenomenon is referred as cavitation or explosive Decompression Failure (XDF) or cavitation [4]. As hydrogen molecules are dissolved in the elastomer amorphous structure, a rapid decompression creates an instability, given that molecules at high pressure can accumulate by diffusion process and generate internal stress. Under adequate conditions, this internal stress can overpass mechanical resistance of the material. Moreover, a major issue of high-pressure containers is that detachment and inside collapse of the liner was observed during discharge, especially at high rate decompressions [6]. This structural collapse is referred as buckling. Partial detachment in localized sectors of the liner is also referred as blistering. Considering that cavitation (at microscopic scale) and buckling (at macroscopic scale) are observed under similar fast depressurisation conditions, recent publications suggest that cavities formation inside the liner might promote buckling failure [7, 8].

We can find bibliography evidence of elastomer failure by cavitation. In the work of Yamabe and Nishimura [5], cavitation is brought to light on pictures coming from transparent specimens or cross-section of o-ring samples, made out of NBR and EPDM. They were submitted to high pressure (up to 90 MPa) for several cycles. This process induced a high level of damage, mainly after several fast depressurisation cycles. Cavitation phenomenon can be observed after depressurisation from high pressure, but also from tensile drawing, which is a simpler way to test cavitation resistance [9, 10].

Moreover, cavitation is reported in thermoplastic materials used as liners for hydrogen tightness, such as polyamides and high-density polyethylene [7, 8, 11]. Samples were pressurised until saturation at pressures ranging from 65 to 90 MPa. After rapid depressurisation, a whitening effect is observed, indicating internal damage, especially at the centre of the samples. Here again, under specific conditions of saturation pressure, depressurisation rate and material thickness, cavitation phenomenon is reported. An increase of internal damage with cycling is also put in evidence [7, 11].

In a previous work, we were able to make a generalization of cavitation risk [12], based on the work of Yersak et al. [8], whom proposed to couple a simplified mechanical failure model with experimental results. We observed that for this model, material's hydrogen diffusivity and yield strength are of utmost importance to evaluate cavitation risk. In present work, we analyse the effect of temperature on these two properties, considering the expected range of operational temperatures of hydrogen containers type IV. As hydrogen diffusivity and yield strength have an inverse evolution with temperature, we studied its effect on our numerical results.

Because of internal energy change of the gas, temperature change is unavoidable to a certain extent in high-pressure systems. We can see from numerical and experimental research articles that high-pressure containers may have internal temperature change up to 80 °C, during both charge and discharge processes [13-16].

2. Numerical model

2.a. Base model

To model the tendency of liner materials to cavitate, we suppose a sample with a geometry that allows dismissing border effects, so that hydrogen diffusion can be considered in one dimension. We considered that time at saturation pressure was enough to obtain a sample fully saturated.

We also considered that transport properties (solubility and diffusivity) are independent of hydrostatic pressure. According to Roger [17], Henry's law is a fair approximation when temperatures are relatively high, and for a low concentration condition. The latter is according to the nature of hydrogen dissolved in polymers, but the former might be a strong supposition. Still, hydrostatic pressure independency cannot be avoided, as there is a notable lack of information concerning transport properties of polymers under high-pressure hydrogen [18]. Generally speaking, bibliography suggests that we can expect a non-linear relation between polymers under hydrostatic pressure and gas diffusivity [19].

To evaluate the evolution of concentration as a function of time and sample depth, we used the following equation [20]:

$$\frac{DC}{kSl^2} = \frac{Dt}{l^2} + \frac{1}{2} \left(\frac{x^2}{l^2} - 1 \right) + \frac{16}{\pi^3} \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n+1)^3} \exp \left\{ -\frac{D(2n+1)^2 \pi^2 t}{4l^2} \right\} \cos \frac{(2n+1)\pi x}{2l} \quad (1)$$

where D is the diffusivity coefficient, S is the solubility of hydrogen, C is the hydrogen concentration, t is the time, l is the half thickness of the sample, x is the depth coordinate. Among possible depressurisation rate regimes [16], we assume a linear one, called k , which can be expressed as follows:

$$k = \frac{(P_{sat} - P_{min})}{t_{deso}} \quad (2)$$

where P_{sat} is the saturation pressure, P_{min} a final minimal pressure at the end of depressurisation, and t_{deso} the time elapsed during depressurisation.

From the approach proposed by Yersak et al. [8], we obtain a parameter called P_{pore} , which quantifies the instability created at the inside of the sample during depressurisation: once the sample is fully saturated, by Henry's law we can estimate the corresponding concentration, which would be identical through all sample volume. Then a depressurisation process will create a concentration gradient inside the sample, which will be more pronounced as rate increases. Assuming pre-existent pores inside the material, P_{pore} considers the difference between pressure resulting from concentration inside the material ($P_{int} = C/S$, according to Henry's law and material's solubility S) and external pressure, which depends on depressurisation rate ($P_{ext} = k.t$):

$$P_{pore} = P_{int} - P_{ext} \quad (3)$$

Finally, a simplified failure model is established, which assumes an elastic field of deformation for the materials under study. It represents the mechanical behaviour of polymers, which present a wide range of strain-stress responses, with viscous, elastic and plastic components. While being a strong simplification, it managed to predict default creation risk in [8], under conditions of interest. Failure model is based on the analyse of the internal pressure of a thick spherical pore. As external to internal radius ratio tends to infinite, a small cavity inside a bulk can be assumed, which is a fair approximation for pre-existing defaults in polymers. Thus, we obtain threshold pressure before arriving to plastic deformation [21]:

$$P_y = \frac{2}{3}\sigma_y \quad (4)$$

2.b. Non-dimensionnel cavitation

Based on the model described above, in a previous work we defined non-dimensional parameters to estimate cavitation risk [12]. Here the notion of cavitation risk implies to obtain a numerical value proportional to the likeness of cavitation to occur. As there remain some strong assumptions (such as constant diffusivity with hydrostatic pressure or purely elastic deformation for polymers), we cannot consider non-dimensional results as deterministic ones, but rather as a tendency of polymers to fail by

cavitation formation. In our previous work we validated numerical results with experimental results found in bibliography.

Non-dimensional parameters consider sample geometry, operational conditions, and materials properties. Cavitation risk is addressed by a parameter named Non Dimensional Cavitation ($NDCav$), according to the following equation:

$$NDCav = \frac{P_{pore}}{P_y} \quad (5)$$

A higher $NDCav$ value involves higher cavitation risk, as pore pressure is higher compared to failure criteria (P_y). $NDCav = 1$ would be the limit to avoid cavitation.

The other two non-dimensional parameters that we proposed to estimate cavitation risk are:

$$T_{deso} = \frac{Dt_{deso}}{l^2} \quad (6)$$

$$M_y = \frac{(P_{sat} - P_{min})}{P_y} \quad (7)$$

Our numerical results show a relation between these three parameters which is consistent with experimental evidence found in bibliography, for semi-crystalline polymers used as liner, and even for elastomers used as o-rings. To briefly picture the core of our previous contribution, we can analyse two cases: i) $NDCav$ values decrease with T_{deso} increasing, which at fixed $(P_{sat} - P_{min})$ condition, is consistent for instance with a lower depressurisation rate (thus, higher t_{deso}), giving more time to hydrogen molecules to diffuse out of the material, minimizing pressure increase inside pores, thus reducing cavitation risk (as in [5, 8]); ii) on the other hand, $NDCav$ values increase with M_y increasing, which implies for instance a higher saturation pressure (P_{sat}) for a fixed minimal pressure (P_{min}), which again is consistent with experiences (as in [5, 10]).

Among the assumption made to evaluate cavitation risk, we supposed constant temperature ($T = 20^\circ\text{C}$) during saturation and depressurisation. In all cases we studied cavitation risk at the centre of the material ($x = 0$) and at the end of depressurisation ($t = t_{deso}$). As cavitation phenomenon is quite

complex, we find that a non-dimensional approach can be useful to evaluate the effect of temperature variation. Further discussion about non-dimensional parameters is found in [12].

2.c. Temperature effect on parameters

In our previous work [12], we observed that material's diffusivity (D) and mechanical performance (yield strength σ_y within P_y) play a major role as input parameters in the non-dimensional analysis. In Figure 1, we show the evolution with temperature of these parameters for high-density polyethylene (HDPE), material taken as reference for calculations, given that there is no glass transition temperature crossing in the temperature range under analyse ($T_g^{HDPE} = -158 \pm 5 \text{ }^\circ\text{C}$ [22, 18]).

Diffusion fitting was obtained considering an Arrhenius equation with the parameters proposed by Humphenoder [23, 18], evaluated at 236 K, 248 K and 298 K, and the values indicated by Yersak et al. [8] at 298 K, 323 K and 358 K. We obtained the following diffusion equation:

$$D(m^2s^{-1})^{HDPE} = 8^{-49} \times T(K)^{15.653} \quad (8)$$

A small extrapolation to 233 K was made to get to the lower limit of temperature range indicated in container's standard.

About hydrogen solubility in HDPE, bibliography information suggest that it is independent of temperature [8, 18] ($S(g_{H2}Pa^{-1}m^{-3})^{HDPE} = 1.01 \times 10^{-5}$).

Yield strength was obtained from linear fitting, as proposed by Merah et al [24]:

$$\sigma_y(MPa)^{HDPE} = -30.15 \times T(K) + 111.86 \quad (9)$$

In this case, extrapolation to the lower limit of the standard was also made, even if implies a relatively large extrapolation. Given that glass transition temperature of HDPE is ≈ 70 K lower than the lower limit, we do not expect any thermodynamic phenomenon that would drastically change the tendency of the curve.

We also made an extrapolation to the higher limit of the standard's temperature range. While extrapolation extension is half in comparison to the lower limit range, we observe that yield strength approaches to zero. However, we accepted this extrapolation values as it represents a conservative approach, and there is no reason to expect a major tendency change. Moreover, we focus our study in understanding cavitation risk evolution with temperature.

Finally, from the work of de Miguel and Zhao [25, 26] we can see that thermal aspects are relevant in type IV hydrogen containers: both fibre composites and thermoplastics are poor heat conductive materials ($\approx 1 \text{ W.m}^{-1}.\text{K}^{-1}$ [18]); temperature change because of container charge or discharge might be relevant (about 40 K in [25, 26]); because of high mechanical solicitations, thick ensembles are expected. Therefore, heat exchange mechanisms will have to be fully evaluated to have an accurate estimation of actual temperature of liner materials. Such study is out of the scope of present work, thus we suppose a homogeneous temperature for the material under analyse, valid for both hydrogen saturation and depletion stages.

3. Results and discussion

3.a. Influence of temperature on cavitation risk for HDPE

To study the influence of temperature in numerical results, we start by fixing the saturation pressure at $P_{sat} = 70 \text{ MPa}$, minimal pressure at $P_{min} = 0 \text{ MPa}$, and we evaluate P_{pore} as a function of material thickness and depressurisation rate. We selected the lowest and the highest temperature values of the hydrogen container's temperature range, and some other values usually taken as reference (i.e.: 298 K). We can see in Figure 2 that the higher the temperature, the lower the P_{pore} parameter (colours are arbitrary). This is consistent with a higher diffusion coefficient, which promotes hydrogen exit of the material, thus diminish pressure inside pores. We consider in all cases $t = t_{desor}$, and $x = 0$.

From the figure, we can also see that there is a region where a saturation pressure is attained, with pore pressure equal to saturation pressure. This would be the case of thicker materials, so as for the higher depressurisation rates. Contrary, for the lower thickness and depressurisation rates under

analyse, we can see that pore pressure remains near zero, meaning that hydrogen gas is able to leave the material by diffusion process with no pressure increase inside pores.

However, in order to consider the effect of temperature in mechanical performance, we re-analyse results of Figure 2 in the form of $NDCav$, by dividing P_{pore} by the P_y value corresponding to the temperature under study (Figure 3). The fact that both thickness and depressurisation rate are independent of temperature allows to have a clear picture of temperature effect on cavitation risk.

Results show that surface-type graphics get cross through the temperature range, meaning that for a certain depressurisation condition, temperature change can have a significant effect on cavitation risk. In other words, results from Figure 3 indicate that the whole temperature range of the technology under study needs to be considered while studying cavitation risk. This figure gives a general picture of the phenomena. To better exploit results, we plot in Figure 4 $NDCav$ vs. depressurisation time for selected sample thicknesses ($2l$), considering width used in references studying liner materials [7, 8, 11]. We kept saturation and minimal pressure values at 70 MPa and 0 MPa respectively. Depressurisation rates of present results are equivalent to depressurisation time range between 24h and 6 min.

From Figure 4a we can see that for present conditions, even for the thinnest sample analysed ($2l = 2$ mm) there is an important restriction in depressurisation rate for the lowest temperature value, in order to remain at a condition of low cavitation risk when $NDCav < 1$, indicated as “No cavitation zone”. As $NDCav$ increases above 1, we observed the appearance of a transition zone, followed by a zone of high cavitation risk, indicated as “Cavitation / transition zone” [12]. From the figure, we also observe that at higher temperatures, cavitation risk remains low. In Figures 4b and 4c we observe that cavitation risk increases with thickness, thus depressurisation rate restrictions become more severe.

Finally, Figure 4d show results for a 10 mm sample, and here we can see crossing lines for the isotherms of lower values. As thickness increases, concentration gradient increases with a quadratic ratio (eq. (1)), thus diffusion phenomenon becomes more important to remove trapped hydrogen before

cavitation occurs. At this state, the exponential relation of diffusivity coefficient with temperature is more relevant than for thinner samples, thus we obtain crossing lines in $NDCav$ calculation.

3.b Effect of temperature in non-dimensional analyse

In this section we evaluate the effect of temperature in non-dimensional analyse, with the aim of knowing if the assumption of constant temperature made in our previous work is relevant or not. We start by analysing P_{pore} (eq. (3)) and its elements. For instance, considering Henry's and 2nd one-dimensional Fick's laws (eq. (1)), and with solubility independent of pressure, it is possible to evaluate internal pore pressure (P_{int}) considering the initial pressure (P_{ini}) and its evolution with time as follows:

$$P_{int}(x, t) = P_{ini} - \frac{P_{ini} - P_{min}}{t_{deso}} \left[t + \frac{x^2 - l^2}{2D} + \frac{16l^2}{\pi^3 D} \sum \frac{(-1)^n}{(2n+1)^3} \exp\left(\frac{-(2n+1)^2 \pi^2 D t}{4l^2}\right) \cos\left(\frac{(2n+1)\pi x}{2l}\right) \right] \quad (10)$$

Therefore, considering that hydrostatic pressure P_{ext} is independent of position, we can re-write equation (3) as:

$$P_{pore}(x, t) = P_{int}(x, t) - P_{ext}(t) \quad (11)$$

Hereafter, a formal mathematical approach shows the non-influence of σ_y and D on $NDCav$ value (eq. (5)), if certain conditions are met. Chosen assumptions correspond to a wide range of cavitation risk evaluation, such as time fixed to t_{deso} , and position is fixed at the centre of the sample ($x = 0$). Time constrain is valid for many applications, for instance for liner material of type IV containers, as a desorption time relatively large permits to obtain maximum pore pressure at the end of container discharge. Finally, sample is considered fully saturated, which implies $P_{ini} = P_{sat}$. Then equation (11) becomes:

$$p_{pore}(0, t_{deso}) = (p_{sat} - p_{min}) \left[\frac{l^2}{2Dt_{deso}} - \frac{16l^2}{\pi^3 Dt_{deso}} \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n+1)^3} \exp\left(\frac{-(2n+1)^2 \pi^2 D t_{deso}}{4l^2}\right) \right] \quad (12)$$

Dividing by the rupture criteria (eq. (4)), and doing replacement considering equations (4), (5) and (6), we obtain that $NDCav$ depends only on non-dimensional parameters M_y and T_{deso} :

$$NDCav(M_y, T_{deso}) = M_y \left[\frac{1}{2} \frac{1}{T_{deso}} - \frac{16}{\pi^3 T_{deso}} \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n+1)^3} \exp\left(\frac{-(2n+1)^2 \pi^2 T_{deso}}{4}\right) \right] \quad (13)$$

Thus, the plot $NDCav$ with respect to M_y and T_{deso} , which perfectly matches Fig. 6 of our previous work [12], is fully independent of yield strength and diffusion. From present model, temperature plays a role in polymers by changing only σ_y and D . This shows that $NDCav$ abacus previously reported is not dependant on the temperature.

4. Conclusions

In present work, we confirm the importance of temperature to evaluate cavitation risk. We observed an overlapping and crossing of $NDCav$ surfaces for different temperatures, as a function of thickness and depressurisation rate. This would be a mix analysis, with a non-dimensional cavitation risk parameter, and two design parameters (thickness and depressurisation rate). From this analysis, we confirm that for the case of HDPE, the most unfavourable conditions of thickness and depressurisation rate will change within containers operative temperature range. For certain conditions, this change can be significant.

On the other hand, non-dimensional parameters are proved to be independent of temperature, which increases the applicability of such analysis. We propose a simplified equation to approximate numerical results previously published.

Nonetheless, while many assumptions were made, and many other parameters remain unexplored (microstructure, cycling, plastic or viscous mechanical effects, etc.), we conclude that present results shall be valid as guidelines to understand cavitation risk tendency for different types of materials, under different operative temperatures.

Acknowledgments

The authors want to thank contributions from mechanical engineering students Pierre Ross and Bay Tangara from ENSMM (École Nationale Supérieure de Mécanique et des Microtechnique, France). This work has been supported by the EIPHI Graduate School (contract "ANR-17-EURE-0002"). Financial

support has been provided by BPI and FEDER from Region Bourgogne Franche-Comté, France (Vhyctor project).

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Figure captions

Figure 1. HDPE hydrogen diffusivity coefficient (diamonds: Humpenoder et al. [23] and squares: Yersak et al. [8]) and yield strength [24] as a function of temperature

Figure 2. P_{pore} for different temperatures as a function of material thickness and depressurisation rate for HDPE ($t = t_{deso}$ and $x = 0$).

Figure 3. Cavitation risk at different temperatures for HDPE.

Figure 4. Cavitation risk vs. depressurisation time for different temperatures for HDPE. Selected thicknesses: **a)** 2 mm, **b)** 3 mm, **c)** 5 mm, **d)** 10 mm.







