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Experimental investigation on charcoal adsorption for cryogenic pump application

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Abstract. Fusion reactors are generating energy by nuclear fusion between deuterium and tritium. In order to evacuate the high gas throughputs from the plasma exhaust, large pumping speed systems are required. Within the European Fusion Programme, the Karlsruhe Institute of Technology (KIT) has taken the lead to design a three-stage cryogenic pump that can provide a separation function of hydrogen isotopes from the remaining gases; hence limiting the tritium inventory in the machine. A primary input parameter for the detailed design of a cryopump is the sticking coefficient between the gas and the pumping surface. For this purpose, the so-called TIMO open panel pump experiment was conducted in the TIMO-2 test facility at KIT in order to measure pumping speeds on an activated carbon surface cooled at temperatures between 6 K and 22 K, for various pure gases and gas mixtures, under fusion relevant gas flow conditions, and for two different geometrical pump configurations. The influences of the panel temperature, the gas throughput and the intake gas temperature on the pumping speed have been characterized, providing valuable qualitative results for the design of the three-stage cryopump. In a future work, supporting Monte Carlo simulations should allow for derivation of the sticking coefficients.

1. Introduction

Fusion reactors are generating energy by nuclear fusion between deuterium and tritium. Very high gas throughputs for the hydrogen species together with helium (a product of the fusion reaction) have to be processed from the plasma exhaust with powerful and high pumping speed systems. Within the European Fusion Programme (EUROfusion), KIT has taken the lead to develop a three-stage cryogenic pump for the next generation demonstration power station DEMO. This concept ranked as fall-back solution only, giving preference to continuously working and non-cryogenic pumps [1-3].

Pumping of light gases relies on cryosorption, the bonding of a gas on a solid surface at low temperature resulting in gas-solid interaction forces stronger than between the particles in a condensed state. Hence, gas can be retained by adsorption in a subsaturated state, at considerably higher temperatures than would be required for condensation. Going towards commercially viable technological solutions, the DEMO reactor will aim at reducing operational costs and demonstrate sufficiently high availability. First of all, the tritium inventory in the fuel cycle of the reactor has to be reduced by implementing a shortcut from pumping to fuelling, by-passing the tritium plant [3,4]. Second, particular attention should be paid on the reduction of the cryogenic operational costs, one of the main drawbacks of cryopumps. These new requirements can be tackled using several pumping stages cooled at different operating temperatures: two pumping stages coated with activated carbon – at 12 K – 22 K for hydrogenic species adsorption and below 5 K for helium adsorption – and tightly surrounded by thermal radiation shields and baffles at about 80 K that also provide a first condensation pumping stage for heavy gases and impurities. By pumping at different stages, this configuration can provide a separation function of hydrogen isotopes from the remaining gases, if the stages are regenerated separately (with regard to time and/or space). Thus, hydrogen can be internally recycled and reinjected. Moreover, the intermediate temperature sorption stage might considerably decrease the heat load to the 5 K cryogenic cooling circuit.

The pumping speed of a cryopump relates to the interaction between the gas and the sorbent which is characterized by the sticking coefficient, the ratio of the number of particles sticking to the sorbent to the number of particles impinging on it; it depends in a complex way upon the gas-adsorbent couple: it decreases with rising temperature of the adsorption surface and rising gas temperature; it decreases with increasing coverage and rising intake gas flow rate and pressure [5]. All these influences make the characterization of the sticking coefficients very complicated and can explain the scarce and inconsistent literature data on that matter. However the sticking coefficient is a primary parameter for the vacuum design of a cryogenic pump, and therefore, its proper characterization is essential. For this purpose, the so-called TIMO open panel experiment was conducted in the large and versatile cryogenic-vacuum TIMO-2 test facility at KIT in order to measure pumping speeds of various pure gases and gas mixtures on an activated carbon adsorption surface, at various sorbent temperatures and under fusion relevant gas flow conditions. Based on the experimental results, a data base of sticking coefficients will be developed later, with the final goal to find the optimum intermediate hydrogen cryosorption pumping stage temperature that offers the best compromise between pumping efficiency and cryogenic operational cost.

This paper summarizes the experimental pumping speed results and the conclusions drawn from this experiment.

2. Description of the TIMO open panel experiment

2.1. Cryopump basic design parameters

The pumping speed of a cryopump is not only given by the sticking coefficient, but also by the interior geometrical arrangement of the pumping surfaces. Both sticking coefficient and geometrical effects are described in an integral parameter indicating the efficiency of a pump: the capture coefficient – the ratio of the pumped gas particles to the total incoming gas particles through the inlet cross section area. As a consequence, the sticking coefficient cannot be obtained directly from experimental results, but it can only be derived from the measurements by comparison with numerical results. Nowadays, predictions for complex geometries are supported by the use of advanced vacuum gas flow modelling tools such as Monte Carlo algorithms [6,7].

2.2. Requirements for the TIMO open panel experimental cryopump

With regard to the very high pumping speeds required in DEMO, the capture coefficient has to be maximized. It can be done within a given volume and with a given pump inlet surface area by increasing the overall molecular transmission probability from the pump inlet to the pumping surface: by (i) opening the inlet baffle’s fins even beyond the limit of optical tightness and (ii) having the adsorbing surface placed on the incoming particles’ mainstream flow path.

It was therefore decided to design the so-called TIMO open panel experiment, consisting of a single adsorption pumping surface coated with activated carbon (cryopanel) loosely surrounded by 80 K thermal shields and having a large inlet cross section area integrating a high transmission probability inlet baffle. This configuration should allow for investigation on two effects which are to be expected under fusion relevant conditions, namely high specific flow rate (related to the pumping surface area) and high gas intake temperature. Moreover, this open configuration results in a capture coefficient (and so a pumping speed) being a strong function of the sticking coefficient, which makes easier the derivation of the sticking coefficient by comparison between the experimental and numerical results.

In order to understand the influence of the gas temperature on the sticking coefficient, it was decided to have a two-phase experimental plan. In the first phase, the pumping surface of the cryopanel is oriented towards the pump inlet, allowing direct hits of particles at ambient temperature on the sorption surface; in the second phase the position of the cryopanel is turned by 180° to have the pumping surface oriented towards the rear side of the pump, hence avoiding the gas particles entering the pump to directly hit the pumping surface without precooling down to the thermal shield temperature (80 K).

2.3. Design and assembly of the test cryopump

The design of the cryopanel aimed at gaining a homogenous temperature distribution over its outer surface. It consists of a stainless steel hydroformed rectangular panel (500 mm x 350 mm) coated with a thin layer of copper and supplied with supercritical helium (SCHe) at 4.5 K. The panel is coated on the center part with activated charcoal (granular coconut shell-based, type SC-II) and bonded with an inorganic cement (THERMOGUSS 2000) over a surface area of about 1220 cm². In order to investigate the influence of the sorbent temperature on the pumping speed, the cryopanel is equipped with resistive electrical wires meandering over the panel surface. The temperature can be regulated between 6 K and 25 K. In order to avoid any potential competitive pumping by condensation on uncoated surfaces, the remaining cryopanel blank surfaces and the 5 K SCHe supply and return lines are insulated with multi-layer insulation material. The panel temperature is controlled and monitored at nine different locations by means of silicon diodes (by Lakeshore, type DT-670-SD).

The external thermal radiation shield of the ITER model pump [8,9] has been reused to surround the cryopanel. It is made of hydroformed dimple plates. The rectangular inlet cross section area (845 mm x 726 mm) integrates a baffle consisting of fins welded on a serpentine cooling pipe. Both systems are cooled with gaseous helium (GHe) at 80 K.

Figure 1 shows a picture of the TIMO open panel cryopump and simplified sketches of the two different pump configurations.

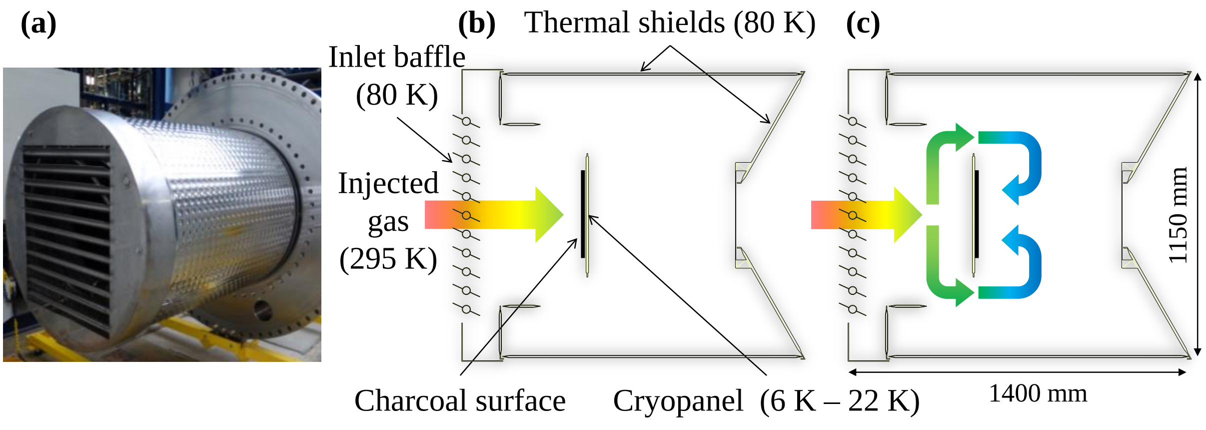

Figure 1. (a) Front view of the TIMO open panel cryopump mounted on its flange; sketches of the two pump configurations: (b) charcoal coated side of the panel oriented towards the inlet baffle; (c) charcoal coated side of the panel oriented towards the rear side of the pump.

2.4. TIMO-2 test facility for large cryopumps

The TIMO-2 test facility [10,11] is a unique test bed for large cryopumps in terms of size and versatility. The gas is metered by thermal mass flow controllers towards the rear dome of the vacuum vessel (~10 m³) designed in accordance with PNEUROP standard for vacuum pump testing. Two pumping stations (roots pump and turbomolecular pump) are connected to the vessel for regeneration and conditioning. The vessel is closed by an adaptable flange used as cryopump support and cryogenic interface. SCHe at 4.5 K and GHe at 80 K are provided by a control cryostat and an 80 K facility. Warm GHe at 300 K and 475 K used for regeneration are provided by external compressors. The valve box distributes helium at different levels of temperature in the two pump circuits (cryopanel and thermal shields/baffle). Two cryogenic flexible lines are connecting the valve box to the pump flange.

Figure 2 shows a picture of the TIMO-2 facility and a CAD view of the TIMO open panel pump installed inside the vacuum vessel.

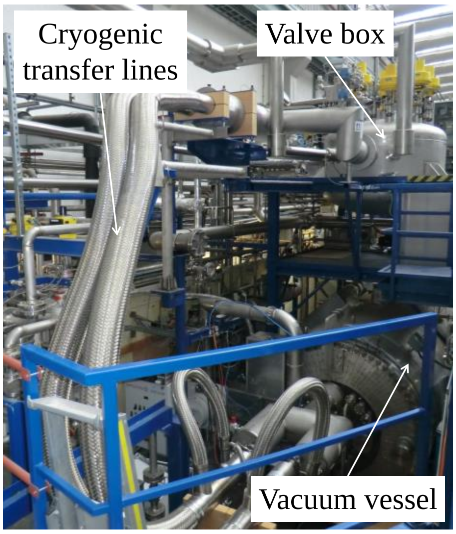

Figure 2. TIMO-2 facility.

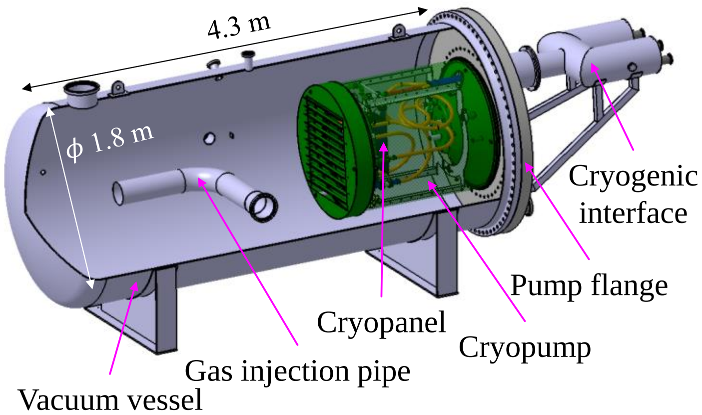

Figure 3. CAD view of the TIMO open panel pump installed inside the vacuum vessel.

The pressure is measured at two different locations: 700 mm in front of the pump, and inside the pump, at the rear side. Each measurement system consists of a cascade of three capacitance diaphragm gauges for measuring pressure from atmosphere down to 10^{-4} mbar and a cold cathode ionization gauge for measurement down to ultra-high vacuum. The pumping speed of the cryopump is given by the gas throughput divided by the pressure in front of the pump. The experimental pumping speeds are given referenced to the standard temperature of 273.15 K.

3. Pumping speed tests with pure gases

During the first experimental phase with the first configuration of the pump (charcoal coated side of the panel oriented towards the pump inlet) several problems occurred with some temperature sensors, making the temperature regulation more complicated and increasing the error on the measured average temperature of the panel. Moreover, the cryopump did not perform very well in comparison with the second configuration (sorption side of the panel oriented towards the rear side of the pump). For these reasons, more effort was done on the second experimental phase, and thus the results presented in this paper mainly focus on the second pump configuration.

Prior to the two experimental phases, the cryopanel and the vacuum vessel were baked out to 150 °C and evacuated simultaneously down to ~10^{-7} mbar by the turbomolecular pump. Partial regeneration at 20 K and 100 K allow for full helium and hydrogen isotopes desorption respectively, whereas the release of air-like species is performed at 300 K. However, the cryopanel was regenerated above 250 K using 300 K GHe prior to each new test with a different gas throughput. Prior to any test with a new gas, both panel and thermal shield/baffle were fully regenerated at 300 K with GHe.

3.1. Tests with pure gases

The pumping speeds of deuterium (D_2), protium (H_2), neon (Ne) and helium (He) have been measured for the two pump configurations. Since He can be pumped efficiently only at temperatures below 7-8 K, all the tests with He have been performed at panel temperature of about 7 K.

ITER plasma scenarios and cryopump operations [12] have been used as a basis to define the gas throughputs to be used for the TIMO open panel experiment. Due to higher gas throughputs in DEMO [3] and limited space for cryogenic pumps, higher specific gas throughputs (related to the pumping surface area) are to be expected. Gas throughputs of 100 sccm, 300 sccm and 500 sccm, corresponding for the TIMO open panel pump to specific gas throughputs of 1.4 (Pa.m³/s)/m², 4.1 (Pa.m³/s)/m² and 6.9 (Pa.m³/s)/m² (at standard temperature of 273.15 K) respectively have been investigated. As the burn fraction of the fusion gas is limited to only few %, additional gas throughputs of 5 sccm, 10 sccm, 30 sccm and 50 sccm have been investigated for He.

Figure 4 shows for the second pump configuration and over 15 minutes the pumping speeds of D_2, H_2 and Ne at panel temperatures between 7 K and 22 K for a gas throughput of 300 sccm, and the pumping speed of He at panel temperature of ~7 K for gas throughputs between 5 sccm and 500 sccm.

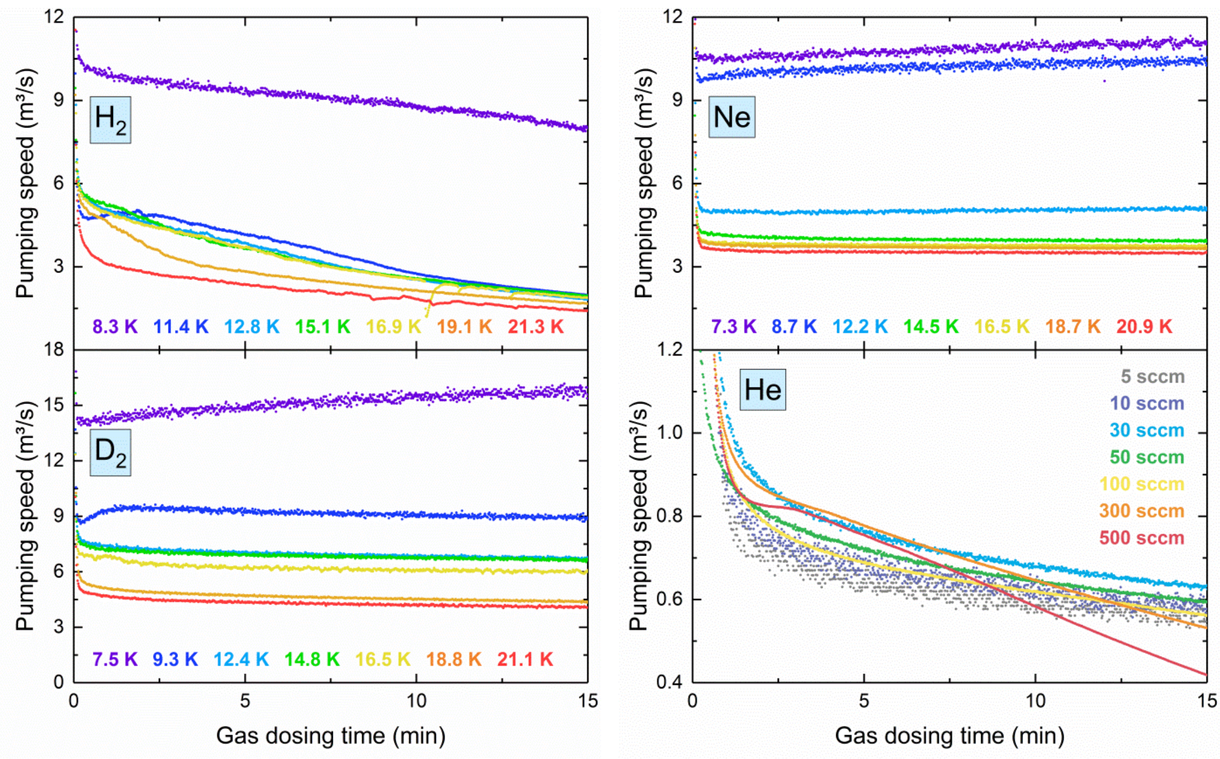

Figure 4. Pumping speeds of D_2, H_2 and Ne over time, at cryopanel temperature between 7 K and 22 K, and for a gas throughput of 300 sccm; pumping speeds of He over time, at cryopanel temperature of about 7 K, and for gas throughputs between 5 sccm and 500 sccm.

Table 1 summarizes for the second panel configuration some average values (panel average temperature *T*, pumping speed *S*, pressure in front of the pump *p* and gas throughput *Q*) calculated over 10 min, a relevant pumping time based on the ITER torus cryopumps' operational mode [12]. Some results with the first panel configuration are additionally presented for comparison. Nonetheless, due to different experimental procedures used in the first experimental campaign, the values shown are not calculated over 10 min of pumping, but at different specific gas load ranges.

The pumping speeds of Ne and D_2 are practically constant whereas the pumping speeds of H_2 and He are decreasing over time, these latter two gases being more sensitive to surface coverage. As long as the charcoal is not saturated, the pumping speeds of all gases increase with increasing gas throughputs. Though, the differences between different throughputs tend to decrease with increasing panel temperature. The increasing pumping speed with increasing gas throughput (and intake pressure) does not mean that the sticking coefficient is increasing, but this is an indication that the pump is operated in transitional pressure regime.

Table 1. Summary of the pumping speed tests with D_2, H_2, Ne and He.

D_2	100 sccm	Configuration 2	*T* (K)	7.3	8.9	12.0	14.5	16.2	18.8	20.6
			S (m³/s)	9.70	6.32	5.38	5.46	4.49	3.60	3.50
			p (Pa)	8.6E-03	2.2E-02	2.6E-02	2.7E-02	3.5E-02	4.7E-02	4.9E-02
		Configuration 1	*T* (K)	6.7	9.0	11.6	14.2	16.4	18.9	21.7
			S (m³/s)	6.35	4.84	4.56	4.23	2.85	2.71	2.45
	300 sccm	Configuration 2	*T* (K)	7.5	9.3	12.4	14.8	16.5	18.8	21.1
			S (m³/s)	14.95	9.25	7.03	6.92	6.25	4.71	4.35
			p (Pa)	2.4E-02	5.2E-02	7.5E-02	7.7E-02	8.7E-02	1.2E-01	1.3E-01
		Configuration 1	*T* (K)	6.5	9.0	11.7	14.3	16.5	18.9	22.2
			S (m³/s)	9.35	6.51	6.21	6.09	3.88	3.76	3.49
	500 sccm	Configuration 2	*T* (K)	8.1	9.4	12.5	15.0	17.1	19.3	21.2
			S (m³/s)	16.25	11.60	7.73	7.59	7.07	4.95	4.74
			p (Pa)	4.0E-02	7.1E-02	1.2E-01	1.2E-01	1.4E-01	2.0E-01	2.1E-01
		Configuration 1	*T* (K)	6.6	9.1	11.7	14.4	16.7	19.1	22.2
			S (m³/s)	10.79	7.10	6.69	6.63	3.96	3.78	3.33
H_2	100 sccm	Configuration 2	*T* (K)	7.6	10.0	12.2	14.5	16.8	19.0	20.7
			S (m³/s)	5.98	3.61	3.36	2.95	2.84	2.55	2.24
			p (Pa)	2.3E-02	4.7E-02	5.3E-02	6.0E-02	6.4E-02	7.3E-02	8.6E-02
		Configuration 1	*T* (K)	6.5	9.9	11.6	14.1	15.7	18.1	21.8
			S (m³/s)	3.26	3.04	2.97	2.04	2.76	1.66	1.41
	300 sccm	Configuration 2	*T* (K)	8.3	11.4	12.8	15.1	16.9	19.1	21.3
			S (m³/s)	9.31	4.00	3.68	3.63	3.58	2.89	2.33
			p (Pa)	5.2E-02	1.5E-01	1.7E-01	1.7E-01	1.8E-01	2.2E-01	2.7E-01
		Configuration 1	*T* (K)	6.3	10.0	11.8	14.3	16.0	18.1	22.0
			S (m³/s)	3.64	3.05	2.92	2.43	2.73	2.12	1.70
	500 sccm	Configuration 2	*T* (K)	8.9	11.0	13.1	15.3	17.4	19.3	21.2
			S (m³/s)	10.10	3.84	2.60	2.95	2.53	2.44	2.08
			p (Pa)	8.7E-02	3.0E-01	4.2E-01	3.7E-01	4.2E-01	4.4E-01	5.1E-01
		Configuration 1	*T* (K)	6.2	10.0	11.9	14.4	16.0	18.6	22.2
			S (m³/s)	3.59	2.79	2.62	2.28	2.42	1.98	1.60
Ne	100 sccm	Configuration 2	*T* (K)	7.0	8.7	11.9	14.3	16.4	18.6	20.9
			S (m³/s)	7.81	6.94	3.49	3.11	2.90	2.92	2.66
			p (Pa)	1.0E-02	1.4E-02	4.6E-02	5.3E-02	5.9E-02	5.9E-02	6.6E-02
		Configuration 1	*T* (K)	6.5	9.7		14.0		18.0	21.9
			S (m³/s)	5.56	2.84		2.23		1.72	1.37
	300 sccm	Configuration 2	*T* (K)	7.3	8.7	12.2	14.5	16.5	18.7	20.9
			S (m³/s)	10.74	10.15	4.99	4.00	3.79	3.71	3.53
			p (Pa)	2.9E-02	3.4E-02	1.1E-01	1.4E-01	1.5E-01	1.5E-01	1.6E-01
		Configuration 1	*T* (K)	6.6	9.8		14.0		18.0	22.0
			S (m³/s)	7.89	4.32		3.31		2.82	2.17
	500 sccm	Configuration 2	*T* (K)	7.6	8.8	12.2	14.6	16.6	18.8	21.0
			S (m³/s)	12.16	12.03	5.68	4.53	4.33	4.17	4.05
			p (Pa)	4.7E-02	5.0E-02	1.6E-01	2.1E-01	2.2E-01	2.3E-01	2.4E-01
		Configuration 1	*T* (K)	6.7	9.8		14.0		18.0	21.8
			S (m³/s)	9.21	5.07		3.86		3.33	2.59
He		Configuration 2	*Q* (sccm)	5	10	30	50	100	300	500
			T (K)	6.8	7.3	7.7	8.2	8.6	8.8	8.7
			S (m³/s)	0.65	0.69	0.77	0.72	0.70	0.77	0.73
			p (Pa)	6.5E-03	1.9E-02	6.8E-02	1.3E-01	3.0E-01	8.0E-01	1.3E+00
		Configuration 1	*Q* (sccm)	5	10	30	50	100	300	500
			T (K)	7.0	7.1	7.2	7.1	6.8	6.7	6.7
			S (m³/s)	0.23	0.23	0.25	0.25	0.25	0.30	0.30

For H_2 and He the surface coverage effects are stronger, and after a certain time, the pumping speeds at the highest gas throughputs become the smallest. As a consequence, when the average pumping speed of He is calculated over a pumping time of 10 minutes, it is practically constant for all the gas throughputs investigated.

From these tests with pure gases, two main conclusions can be drawn. First, the issue raised concerning potential strong negative effect of higher gas temperature on the sticking coefficient was sound: it is clear that the charcoal coated surface area being now protected from gas at high temperature directly impinging it, the pumping performances of the TIMO open panel with the second configuration are significantly improved, particularly at the lowest panel temperatures and for H_2 and He (pumping speed increased up to a factor ~3 for He), the two lightest and most difficult gases to pump. Second, the pumping speed of D_2, H_2 and Ne is rapidly decreasing between panel temperatures of 7-8 K and 12 K, and more slowly between 12 K and 22 K. The conclusion drawn from this statement is that having an intermediate hydrogen pumping stage at ~20 K could be more efficient than at 12 K in terms of pumping performance over cryogenic cost.

3.2. Tests with hydrogen-helium gas mixtures

The pumping speeds of six different hydrogen-helium gas mixtures have been measured with the second pump configuration in order to study the impact of the He concentration on D_2 and H_2 pumping. Figure 5 shows the average pumping speeds over 15 minutes of D_2-He and H_2-He gas mixtures as well as D_2, H_2 and He pure gases, at panel temperature of about 7 K and for gas throughputs between 10 sccm and 500 sccm.

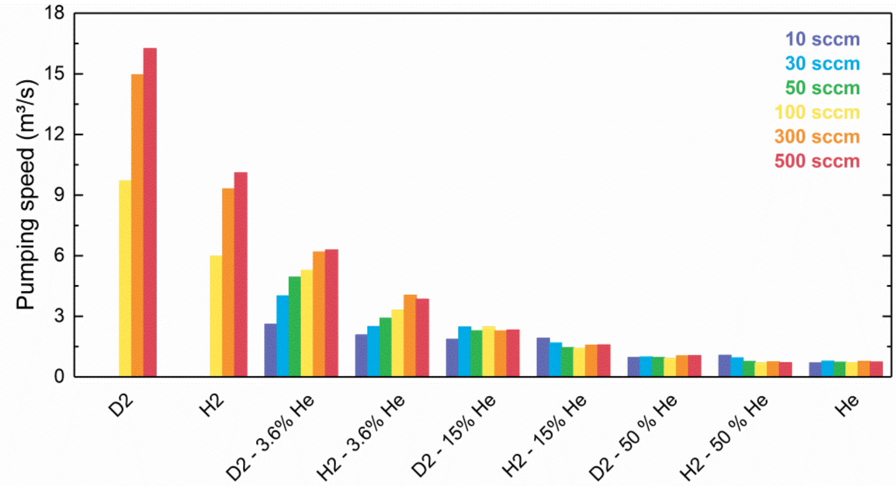

Figure 5. Average pumping speeds of D_2, H_2, He, D_2-He and H_2-He gas mixtures over 10 minutes of pumping at panel temperature of about 7 K, for gas throughputs between 10 sccm and 500 sccm.

The main conclusion of these tests is that hydrogen and He are strongly competing for sorption sites on the carbon surface and thus the pumping speed of D_2 and H_2 is exponentially decreasing with the fraction of He into the hydrogen mixtures. Similar conclusion was drawn from an older experiment at KIT [13]. The burn fraction in DEMO is consequently a primary important parameter for the three-stage cryopump design.

4. Conclusion and outlook

Within EUROfusion, a three-stage cryopump has to be developed for DEMO. The first step of the development related to the characterization of the interactions between the gas and the sorbent – at different sorbent temperatures and for different fusion relevant gases (D_2, H_2, He and Ne) and flow conditions – has been successfully achieved with the so-called TIMO open panel experiment. This experimental work included two phases using two different geometrical configurations of the cryopanel, allowing for a deeper understanding of the influence of some key parameters on the sticking coefficient. In particular, this experimental plan was aimed to investigate two effects which are to be expected under fusion relevant conditions, namely high specific flow rate related to the charcoal surface area and high gas intake temperature.

The TIMO open panel pump performed significantly better with lower intake gas temperature, and it is now clearly confirmed that higher temperature of the incoming gas to the cryopanel is strongly harming the pumping performance. Thus, one of the main objectives of this task was achieved. Another conclusion is that having an intermediate hydrogen pumping stage at about 20 K could be more efficient than at 12 K in terms of pumping performance over cryogenic cost.

Additionally to the test with pure gases, co-pumping effects have been investigated with various hydrogen-helium mixtures. The pumping speed of D_2 and H_2 is exponentially decreasing with the fraction of He into the mixtures, so that at He concentration above 15% the hydrogen species practically does not play a role anymore. Hence, the burn fraction in DEMO will be a primary importance parameter for the design of the three-stage cryopump.

Support Monte Carlo simulations have been initiated in order to derive the sticking coefficients by comparison with the numerical results. Based on the experimental and numerical results, a data base of sticking coefficients at different sorption cryopanel temperature and various gas flow conditions – to be used in future numerical simulations during the vacuum design phase of the three-stage cryopump – will be developed, with the final goal to find the optimum intermediate hydrogen cryosorption pumping stage temperature.

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