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Thermal behaviour of ZrCo getter beds during hydrogen absorption at variable flow rates and concentrations

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ABSTRACT

Zirconium-cobalt alloy (ZrCo) is one of the most studied getter materials for the storage, supply, and recovery of hydrogen isotopes. However, the exothermic nature of the hydridation reaction has always raised concerns regarding local overheating, which might compromise the getter stability, particularly at high H-concentrations and/or high flow rates. In this work, the thermal behaviour of a ZrCo getter bed upon hydrogen absorption was experimentally assessed under controlled conditions in the HYDE loop facility installed at the Tritium Laboratory Karlsruhe (TLK). Successive loading–deloading cycles were performed on 30 g of ZrCo with H_2/He mixtures ranging from 1 % to 11 % H_2 under both low (40–100 Ncm^3/min) and high (300–500 Ncm^3/min) H_2 flow regimes. A linear relationship ($R^2 \approx 0.99$) was observed between the H_2 concentration in helium and the maximum temperature reached by the getter material during absorption, regardless of the helium dilution rate. The temperature increased from 68 °C at 1 % H_2 (low flow) to 258 °C at 11 % H_2 (high flow). The reproducibility of the experiments in terms of absorption capacity with nearly 40 experimental cycles also confirms that the material remains stable, with an average H/ZrCo ratio of $\sim 2.80 \pm 0.06$. These results show that under DEMO-relevant operating conditions (<0.1 % H_2 in helium), the peak temperature that can be reached by the ZrCo getter should be moderate, and it is not expected to be a matter of concern or to jeopardise the getter material stability.

1. Introduction

Hydrogen isotope control is of great importance in fusion technology since it is required to be linked to safe and efficient tritium storage, recovery and supply systems. In a context of multiple candidate getter materials, zirconium–cobalt (ZrCo) is by far the most studied alloy and has thus become the reference material for the handling of hydrogen isotopes, in terms of absorption/desorption kinetics, capacity and cycling behaviour [1–3]. However, some issues remain on ZrCo stability under particular operating conditions. In particular, ZrCo suffers from a disproportionation mechanism at temperatures above 400 °C. Disproportionation is degrading the gettering properties of ZrCo because it is converting $ZrCoH_3$ to convert into ZrH_2 and ZrCo, and thus show a significant loss of its gettering properties. Disproportionation is a major drawback as it fixes almost irreversibly part of the hydrogen (hence, tritium) into a ZrH_2 form [4–7] .

More recently, it has been reported that the heat released during the exothermic absorption reaction can itself cause very significant local temperature rises in the getter bed [8]. These thermal excursions have a significant impact on both the integrity of the material and the retention of the isotopes, even more so when tritium (T_2) is concerned.

As a matter of fact, the previously cited simulation study showed that the hydridation of ZrCo can lead to local bed temperatures rising by several hundreds of degrees [8]. To date, however, experimental data on the temperature evolution during hydrogen absorption under controlled flow conditions are very scarce, and systematic studies are required. The present study aims at filling this gap, which is a prerequisite to the design of getter systems for ITER Test Blanket Modules (TBMs) and future power plants like DEMO.

In this paper, an experimental investigation to thermally characterise ZrCo getter beds during H_2 absorption at different flow rates and H_2 concentrations has been reported. The measured outcome of the study is an attempt to correlate gas composition and flow conditions to resulting temperature rise in order to evaluate temperature-wise the operational safety margin of the ZrCo based getter technology.

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2. Experimental

Commercial ZrCo powder was obtained from SAES Getters (Milan, Italy). The material was supplied as a single batch in a coarse powder form, which suggested it had undergone previous hydrogen cycling. The material was activated before testing by heating under moderate vacuum (1E-4 mbar) to 420 °C for several hours in order to release the residual hydrogen.

Experiments were performed on the HYDE (HYdrogen-DEuterium) loop at KIT [9,10], which was originally built for isotopic calibration and has since been adapted for getter-bed testing. The loop is fitted with mass flow controllers for hydrogen and helium, quadrupole mass spectrometry (QMS) and a number of pressure and temperature sensors. The addition of a further buffer vessel increased the overall volume of the loop to 10.55 L and allowed the full loading of the getter bed.

The getter bed was a stainless-steel tube divided in six compartments (Fig. 1) in order to reduce clumping of the material due to the volume expansion of the hydride (~23 % volume change from ZrCo and $ZrCoH_3$). Not accounting for the volume expansion of ZrCo resulted in damage to the structure of the getter bed because the seals degraded, causing gas leaks [11]. It has an inner diameter of 42.5 mm and a length of 148 mm and in the present experiments 30 g of ZrCo was loaded into the central compartment. A thermocouple was placed directly into the getter material to record the in-situ temperature. Porous Siperm R3 stainless steel discs were placed before and after the getter bed in order to contain the getter material and prevent the release of the smaller particles into the rest of the experimental hardware, and at the same time minimising the pressure drop.

An in-depth explanation of the entire system, including the HYDE facility, the getter bed, and its various components, has been reported elsewhere

Two campaigns were performed.

- Low-flow regime: 40–100 Ncm^3/min H_2 with He carrier gas (H_2 fractions: 1–10 %).
- High flow regime: 300–500 Ncm^3/min H_2 with He carrier gas (H_2 fractions: 1.7–11 %).

The inlet/outlet pressures, the flow rates, the bed temperature, and the amount of absorbed hydrogen were recorded in each measurement. Deloading was performed between the cycles to ensure reproducibility.

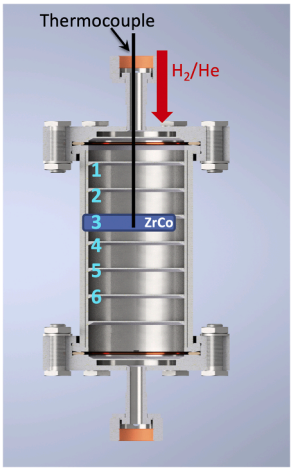

Fig. 1. Schematic illustration of the getter bed as in operation.

3. Results

3.1. Low flow regime (40–100 Ncm^3/min)

The first series of measurements for the low-flow regime is presented in Fig. 2 (bottom line).

As it may be seen, at relatively low H_2 flow rates, the maximum bed temperature increased linearly with the hydrogen fraction in He. The temperatures varied from 68 °C (1 % H_2) to 134 °C (10 % H_2), while the volume of absorbed hydrogen was nearly the same for all the tests (~6.6–6.8 NL) corresponding to atomic ratio H/ZrCo ≈ 2.77. It should be noted that the absorption capacity was not degraded after several load/unload cycles.

3.2. High flow regime (300–500 Ncm^3/min)

The second series of measurements (high-flow regime) results is presented in Fig. 2 (top line).

As it may be seen, the effect of higher H_2 flow rates is evident, and much higher peak temperatures are registered. At 1.7 % H_2, the peak value of ~194 °C was measured in comparison with ~76 °C under low-flow 2 % conditions. Moreover, by further increasing the hydrogen fraction, the temperature has increased up to 225 °C (5 % H_2), 232 °C (7.5 % H_2) and 257 °C (11 % H_2). As it may be clearly seen, both the H_2 fraction and H_2 flow rate had an influence on the thermal response.

Both the low-flow (40–100 Ncm3/min H_2) and the high-flow (300–500 Ncm3/min H_2) regimes presented a strong linear correlation between the hydrogen fraction and the maximum bed temperature with a very good coefficient of correlation (R^2 ≈ 0.99) for both regimes.

For example,

$$\text{Low} - \text{flow fit} : \text{Peak(temp)} \approx 58.7 + 8.14 \times (\%\text{H2}) \quad (1)$$

$$\text{Low} - \text{flow fit} : \text{Peak(temp)} \approx 183.6 + 7.40 \times (\%\text{H2}) \quad (2)$$

The comparable slopes (~7.4–8.1 °C per % H_2) imply that the peak temperature is governed mainly by the H_2 concentration in the gas mixture (reaction enthalpy scales with H_2 partial pressure). Additionally, the variation of Helium flow rate had no notable effect on peak temperatures. In other words, there was almost no cooling by the addition of higher amounts of He.

In addition to the correlation between H_2 flow rates and peak

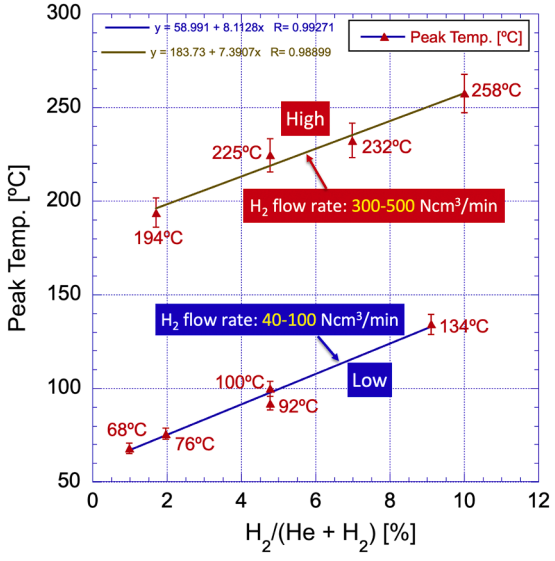

Fig. 2. Summary of the results illustrating the relationship between the peak temperature increase and the H_2 fraction in the purging He gas. The bed is loaded either with low or high flow rates of H_2.

temperature, we should also note that with a higher flow, the corresponding loading times were shortened (13–22 min). This observation clearly indicates the existence of a kinetic mechanism as the duration of absorption time has decreased proportionally with an increase of gas flow rate. Table 1 summarises the high-flow and low-flow results obtained for H_2 concentration ranging between 1 and 11 %.

4. Discussion

Our results have shown that the thermal behaviour of ZrCo during hydridation is strongly dependent on both the hydrogen concentration in the He carrier gas and the hydrogen flow rate. Whether by increasing the H_2 concentration in He or by increasing the H_2 flow rate, the results is an increase of the amount of hydrogen arriving at the getter bed. This leading to the acceleration of the H_2 absorption reaction. This in turn, will lead to higher peak temperatures since more heat is generated.

As it is illustrated in Fig. 2, the two flow regimes are nearly parallel having similar slopes. However, compared to the low-flow results, the high-flow line is shifted upward. This is indicating that for any given % H_2, the getter material becomes hotter when the H_2 gas flow is increased.

Moreover, if for the same H_2 concentration, we compare the two linear fits we may also note that each time the higher temperature is obtained for the higher flow rate, although for both flow regimes the thermodynamic heat of absorption is the same. The reason for this upward shift of the high-flow rate regime is due to the fact that a greater number of H_2 molecules arrive at the getter bed per unit time, which results in more heat produced within the same timeframe. Obviously, because the heat generation rate is related to the H_2 molar fraction entering into the getter bed, an increase in the amount of H_2 reaching the getter bed leads to a more significant temperature increase.

It is also worth mentioning that the intercepts of both linear fits towards the y-axis are simply empirical baseline offsets, that characterise each flow regime. This intercept does not represent the literal temperature of the getter bed at 0 % H_2, since there is no reaction when there is no hydrogen. These intercepts rather reflect the vertical adjustment that the regression analysis requires in order to be aligned with the data obtained within our measured range of H_2 percentages.

Also, if we consider the maximum temperatures obtained for the same H_2 concentration for the two flow regimes, we realize that the upshift of the high-flow curve also indicates that heat removal does not seem to be very relevant. In the context of packed getter beds, heat removal is usually done by conduction through the solid matrix to the walls and surroundings and is not by gas convection. In fact, at low/moderate velocities the hydrogen/helium flow rate usually has a low heat capacity rate, and lower heat-transfer coefficients, with respect to solid conduction. So, although an increase of the total flow slightly increases heat transfer by gas convection, it usually plays a minor role.

Another observation that is worth to note is that during these experiments, the maximum peak temperature for ZrCo does not exceed relatively low values. For instance, even under high H_2 concentration (up to 10 %) and high flow rates (up to 500 Ncm^3/min) maximum temperature reached in the bed does not exceed 258 °C and working with 100 % H_2 at 5NL/min maximum temperature does not go above 320 °C, which well below 400 °C threshold associated with disproportionation of ZrCo [12]. This fact suggests that for DEMO operation we have a very good safety margin to avoid disproportionation working with ZrCo under well controlled operation conditions (for pressure and temperature). Indeed, for DEMO operation conditions, the hydrogen concentration in helium purge streams is expected to be approximately 0.1 % or lower [13]. Under such conditions and referring to Eqs. (1) and (2), the temperature rise of the getter bed will hardly exceed 59 °C for the low-flow rate and 184 °C for the high-flow rate.

Another important note is that the material has a very strong resistance to repetitive load-deload cycling. For instance, even after >40 loading and deloading cycles using the same sample of ZrCo, the loading capacity remains practically unchanged and is equal to $x = 2.80 \pm 0.06$ where x is the atomic H_2/ZrCo ratio. This fact is also in favour of ZrCo for long term applications in tritium-handling systems.

All data available for both regimes (high-flow and low-flow) are summarized Fig. 3. As it can be seen, an exponential relationship fits both regimes very nicely, while the two linear fits give an approximate picture of the reaction each one corresponding to a specific flow regime.

The exponential relationship suggests that higher peak temperatures are associated with shorter times to reach those temperatures. That implies that as the peak temperature increases, the time to reach that temperature decreases rapidly at first and then levels off.

The reason why the exponential fit describes better what happened is related to the fact that the process is reaction/absorption-rate controlled with Arrhenius-like kinetics i.e. the absorption reaction rate is a function of the temperature; $k(T) \approx k0 \cdot \exp(-Ea/RT)$. In other terms, as the getter heats up from the exothermic H_2 absorption, the local reaction rate

Table 1
Summary of the high-flow and low-flow results obtained for H_2 concentration ranging etween 1 and 11 %, including the experiment with pure H_2.

Low-Flow rates 40–100 Ncm^3/min					
H_2 / ($He+H_2$)	H_2 Flow Rate	He Flow Rate	Peak Temp.	Time to Peak temp.	Abs. H_2
[%]	[Ncm^3/min]	[Ncm^3/min]	[°C]	[min]	[NL]
1	55	5500	68.0	(79)	6.58
2	40	2000	75.8	144	6.45
4.8	55	1100	92.2	106	6.66
4.8	55	1100	99.9	111	6.63
9.1	100	1000	134.3	64	6.73 <x = 2.77>*
High-Flow rates 300–500 Ncm^3/min					
H_2 / ($He+H_2$) [%]	H_2 Flow Rate [Ncm^3/min]	He Flow Rate [Ncm^3/min]	Peak Temp. [°C]	Time to Peak temp. [min]	Abs. H_2 [NL]
1.7	300	17,500	193.9	29	6.8
4.8	400	8000	224.5	20	6.90
7.0	375	5000	232.4	37	6.85
10.0	500	4500	257.5	17	6.71 <x = 2.85>*

*where x is the atomic H/ZrCo ratio.

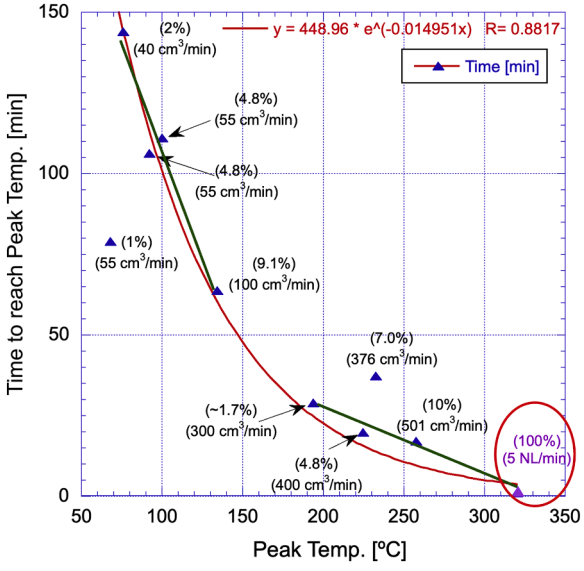

Fig. 3. Summary of the results illustrating the relationship between the peak temperature increase and the time needed to reach it.

accelerates roughly exponentially with temperature, so the time it takes to "finish" rising to its peak shrinks rapidly giving the phenomenological appearance of an exponential decay.

As temperature rises, the kinetic barriers (dissociative adsorption, surface diffusion, bulk diffusion, hydride nucleation/growth) are overcome faster, so k(T) grows approximately exponentially with T (in absolute temperature).

The reaction is exothermic and self-heating

- Each increment of H absorbed releases heat, raising the solid temperature.
- Higher T → higher k(T) → more heat generated per unit time → higher T.

This positive feedback accelerates the climb to peak temperature.

5. Conclusions

The present work provides a systematic investigation of the thermal response of ZrCo during hydrogen absorption under controlled flow and concentration conditions. The experimental results have shown that there is a strong linear relationship ($R^2 \approx 0.99$) between the hydrogen concentration in the carrier gas (He) for both the low-flow and high-flow regimes.

In both cases, the peak temperature was directly related to the amount of hydrogen arriving at the getter bed, whether by increasing the H_2 concentration in He or the total gas flow rate. The highest peak temperature (~ 258 °C) was obtained when we worked with the highest hydrogen concentration (10 %) in the high-flow-rate regime (500 Ncm^3/min). This difference in peak temperature compared to the low-flow regimes is explained by the increased number of H_2 molecules arriving at the getter bed per unit time. Moreover, the maximum peak temperature (~258 °C) observed for the H_2/He gas mixture is still well below the 400 °C threshold for ZrCo disproportionation. Even when working with 100 % H_2 at 5NL/min the peak temperature did not reach >320 °C. Therefore, we may reasonably assume that under DEMO-relevant operational conditions, where the hydrogen fraction in helium purge gas is expected to be below 1 %, the corresponding temperature rise that can be expected will hardly exceed 70 °C. This confirms that ZrCo getter systems can operate safely within the required thermal margins.

The material also exhibited outstanding stability and reproducibility throughout the entire series of experiments. After approximately forty (40) absorption–desorption cycles, it maintained a consistent hydrogen-to-metal ratio of approximately 2.8. This observation also confirms that working under well controlled conditions the material can consistently absorb the same amounts of H_2 while simultaneously its degradation due to the disproportionation reaction is prevented or limited.

Finally, kinetic analysis revealed that the relationship between peak temperature and absorption time follows an exponential trend, which is consistent with an Arrhenius-type reaction rate.

Overall, the present results have shown that ZrCo getter beds are reasonably stable when working under controlled conditions, exhibiting a safe thermal performance under a wide range of hydrogen flow and concentrations, reinforcing their suitability for tritium storage and recovery applications in ITER and DEMO fusion systems.

Recommendations

For hydrogen cycling, ZrCo is thermally sturdy and typically operates effectively in the range of 200–400 °C. The peak temperatures (up to 258 °C) observed during absorption of low H_2 concentrations (up to 11 %) or even for pure H_2 (320 °C) are well below 400 °C above which disproportionation is taking place. Therefore, for DEMO where the He stream will be hardly loaded with >1 % H_2 the getter will hardly reach >70 °C.

However, for high-throughput applications requiring 300–500 Ncm3/min, in order to better manage the thermal excursions generated by the fast hydrogen absorption, it is recommended to enhanced heat sinks inside the getter bed (e.g. using fins) or foresee an active cooling of the getter bed.

Finally, by using a gradual loading of the getter bed, we will allow a production of small quantity of heat thus avoiding too high peak temperatures. Also, by making some adjustments to the size and shape of the getter bed, such as include some fins, will improve the heat transfer to the walls. It will be also worth considering to distribute the getter material into separated sections or compartments within the getter bed. This will also help with the heat dissipation.

CRediT authorship contribution statement

Nicolas Bekris: Writing – review & editing, Writing – original draft, Validation, Supervision, Software, Methodology, Formal analysis, Conceptualization. **Monica Sirch:** Data curation. **Robin Größle:** Supervision, Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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