Control of excess heat production in Pd-impregnated alumina powder

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Abstract

We carried out an experimental study of excess heat production during deuterium loading of Pd-impregnated alumina. Earlier studies [1,2] have shown that a hydrogen-deuterium (H/D) exchange chemical reaction can account for at least some of excess heat observed during gas-loading experiments. In this work we show that excess heat contributed by H/D exchange can be eliminated by prebaking the material in vacuum at 390ºC, due to the removal of residual water from the material. After the material is given the opportunity to reabsorb water from air the reaction and excess heat production in the presence of deuterium resumes. Our calculations on the energy available from H/D exchange show that all the excess heat observed during our experiment can be accounted for by this chemical reaction.

[1] D. Kidwell, D. Knies, A. Moser, D. Domingues, "*Yes, Virginia there is heat, but it is most likely of chemical origin,*" Proceedings of the 15th International Conference on Condensed Matter Nuclear Science, (2009) 100-109

[2] O. Dmitriyeva, R. Cantwell, M. McConnel, and G. Moddel, "*Mechanisms for heat generation during deuterium and hydrogen loading of palladium nanostructures,*" Proceedings of the 16th International Conference on Condensed Matter Nuclear Science, (2011) in press

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Motivation

- Excess heat in Pd-impregnated materials with D_2 [1-5]
- Evidence of exothermic H/D exchange chemical reaction in water[4,5]

[1] Y. Arata, Z. Chang, Establishment of the "Solid Fusion" reactor, J. High Temp Soc., 34 (2008) 85-93.
[2] Y. Sasaki, A. Kitamura, Y. Mioshi, T. Nohmi, A. Taniike, A. Takahashi, R.Seto, Y. Fujita, Proceedings of ICCF15, (2009) 94-99.
[3] T. Hioki, H. Azuma, T.Nishi, A. Itoh, J. Gao, S. Hibi, T. Motohiro, J. Kasagi, Proceedings of ICCF15, (2009) 88-93.
[4] D. Kidwell, D. Knies, A. Moser, D. Domingues ICCF15, (2009) 100-109.
[5] O. Dmitriyeva, R. Cantwell, M. McConnell, and G. Moddel, ACS National Meeting and Exposition, Anaheim, 2011

Motivation

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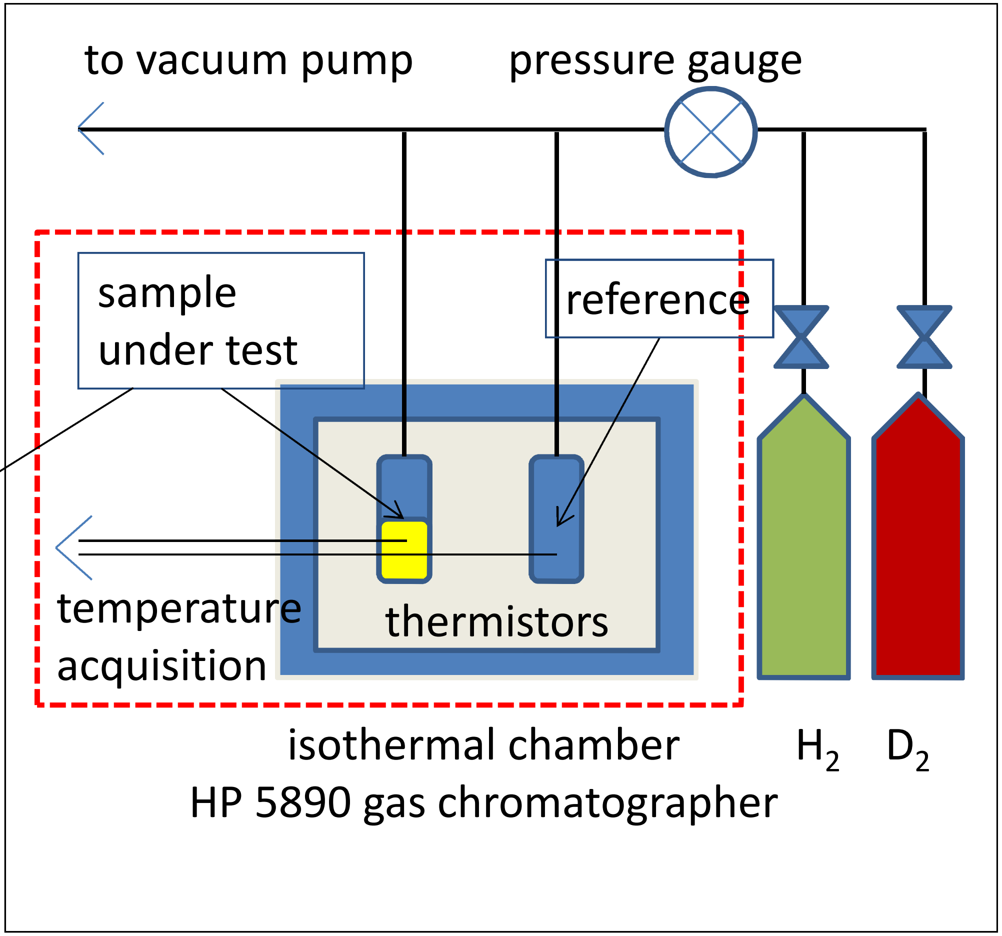
- Evidence of H/D exchange chemical reaction in water[4,5]

Questions to answer

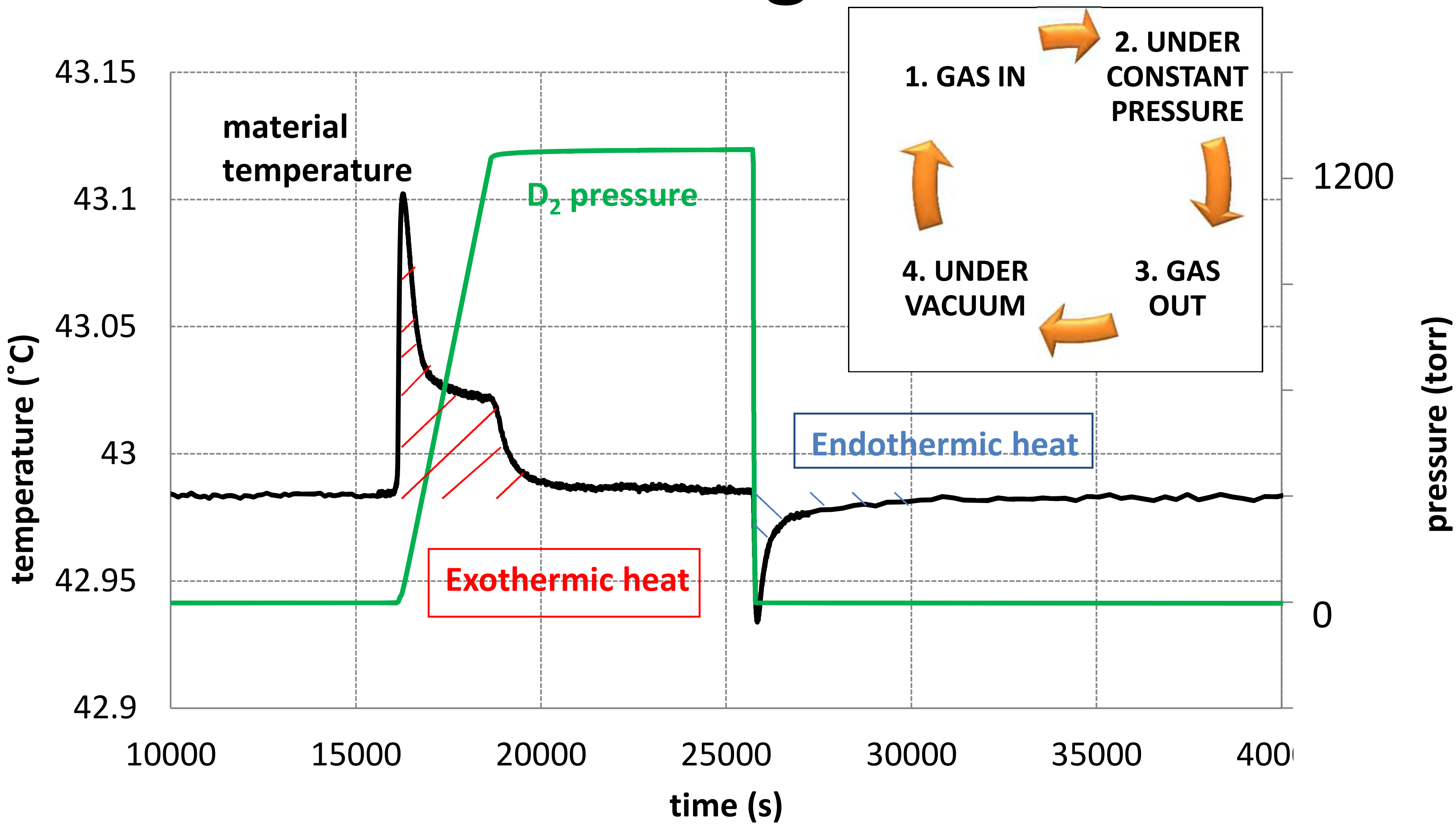
- Control excess heat by controlling amount of water?
- Enough water to account for excess heat?

Gas-loading system

- Precision of temperature control 10 mK
- 6 g of material
- Pressurized up to 1200 torr
- Temperatures 40 - 390°C



Excess heat generation



Excess heat = Exothermic heat + Endothermic heat

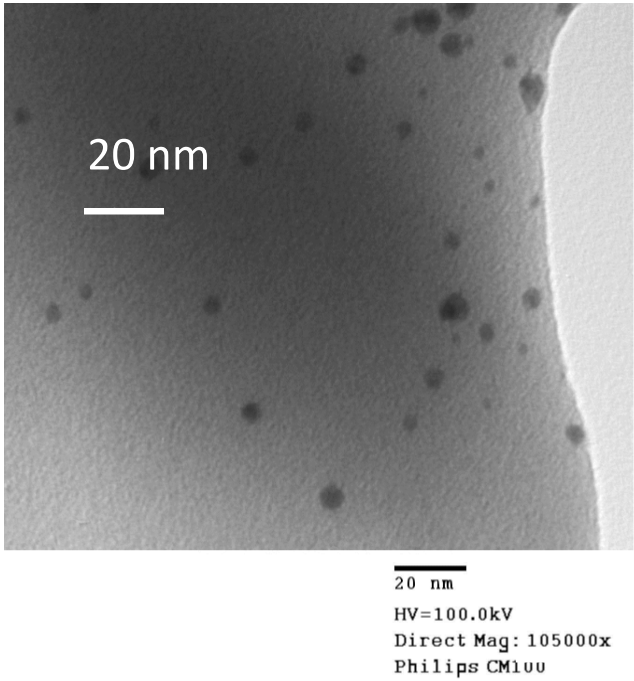
Material fabrication

- Oxide supports:
  - alumina: Fisher Scientific P/N: CAS 1344-28-1
    74-177 µ particle size
  - high crystalline α-alumina: American Elements
    P/N AL-OX-O2-P.30UM 30-50 µ particle size

- 2% Pd by weight in by wet impregnation of different precursors:
  - $Pd(NH_3)_4Cl_2$
  - H_2PdCl_4

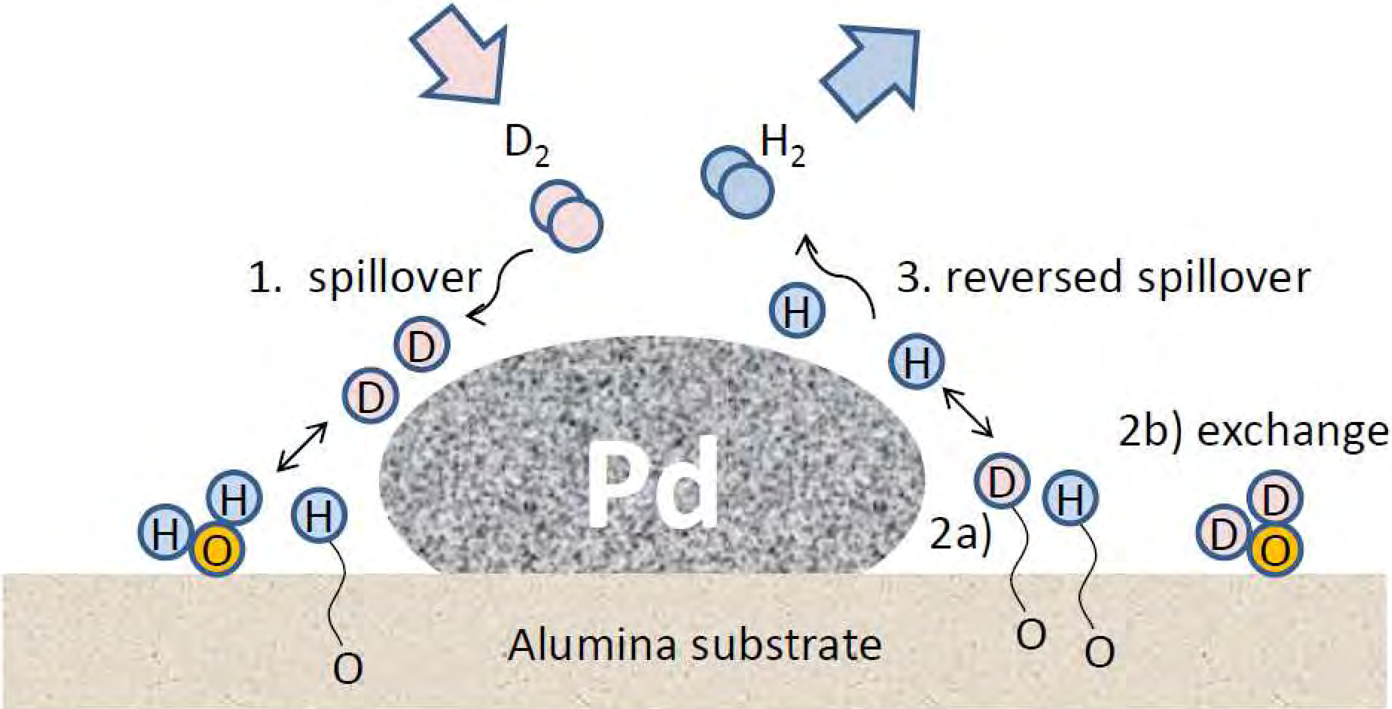
commercially available 5% Pd in alumina Acros Organic CAS:7440-05-3

average particle size 5 nm



O. Dmitriyeva, R. Cantwell, M. McConnell, and G. Moddel, “Mechanisms for heat generation during deuterium and hydrogen loading of palladium nanostructures”, ICCF-16, Chennai, India, 2011, in press

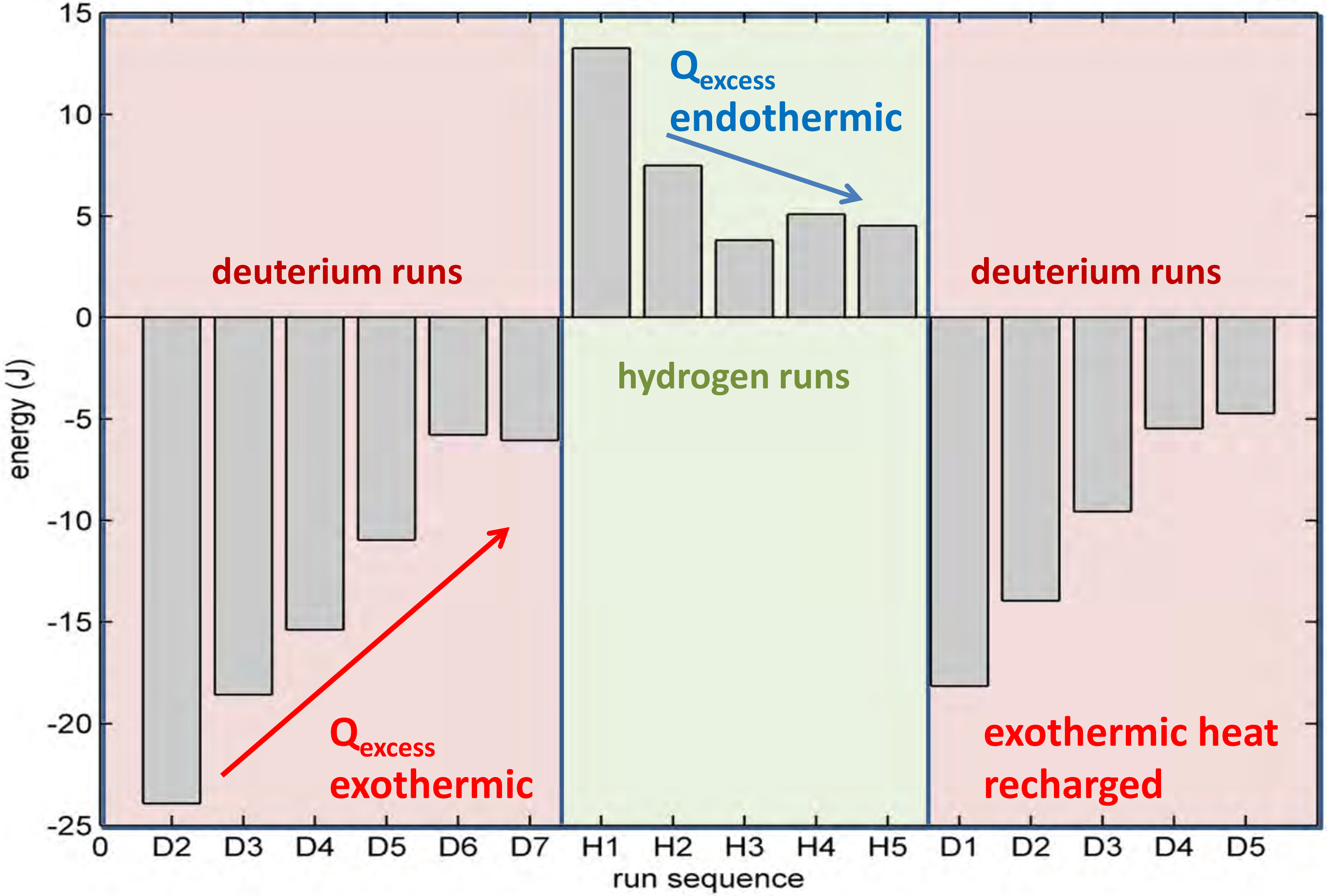
H/D exchange reaction



OH + D -> OD + H (- 4.0 KJ/mol)	OD + H -> OH + D (4.0 KJ/mol)
H_2O + 2D -> HDO + HD (- 4 KJ/mol)	D_2O + 2H -> HDO + HD (4.3 KJ/mol)

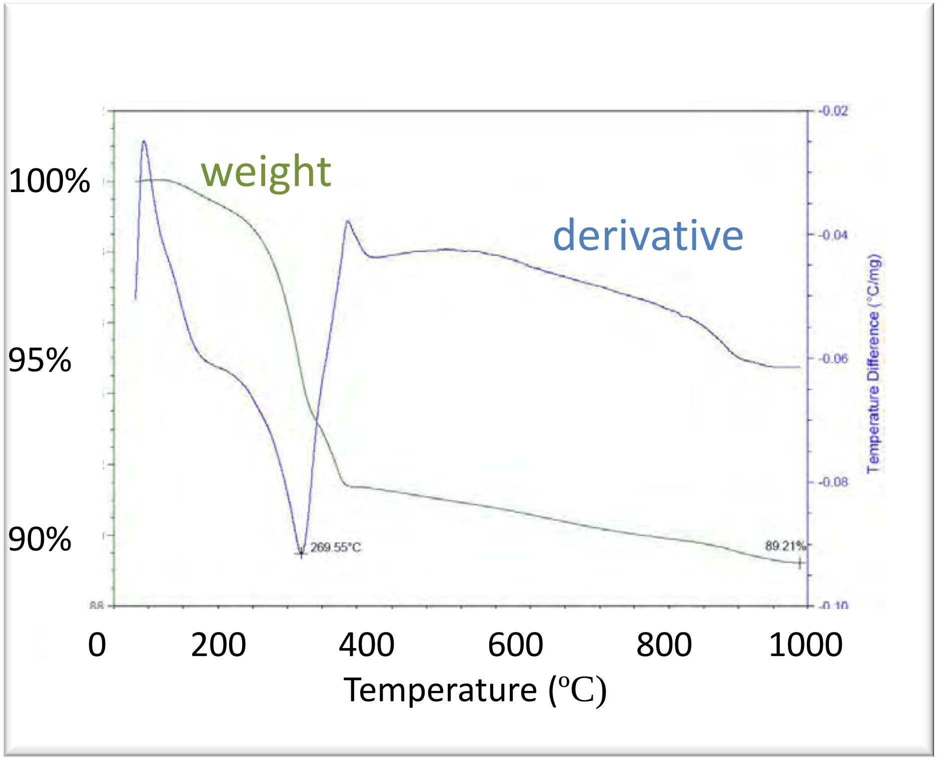
D. Kidwell, D. Knies, A. Moser, D. Domingues, "Yes, Virginia there is heat, but it is most likely of chemical origin", Proceedings of the 15th International Conference on Condensed Matter Nuclear Science, (2009) 100-109.

Heating and cooling due to H/D exchange



Presence of water

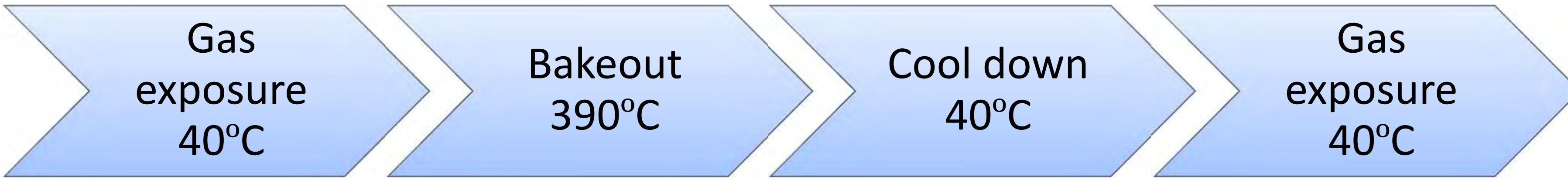
Thermogravimetric analysis



Summary

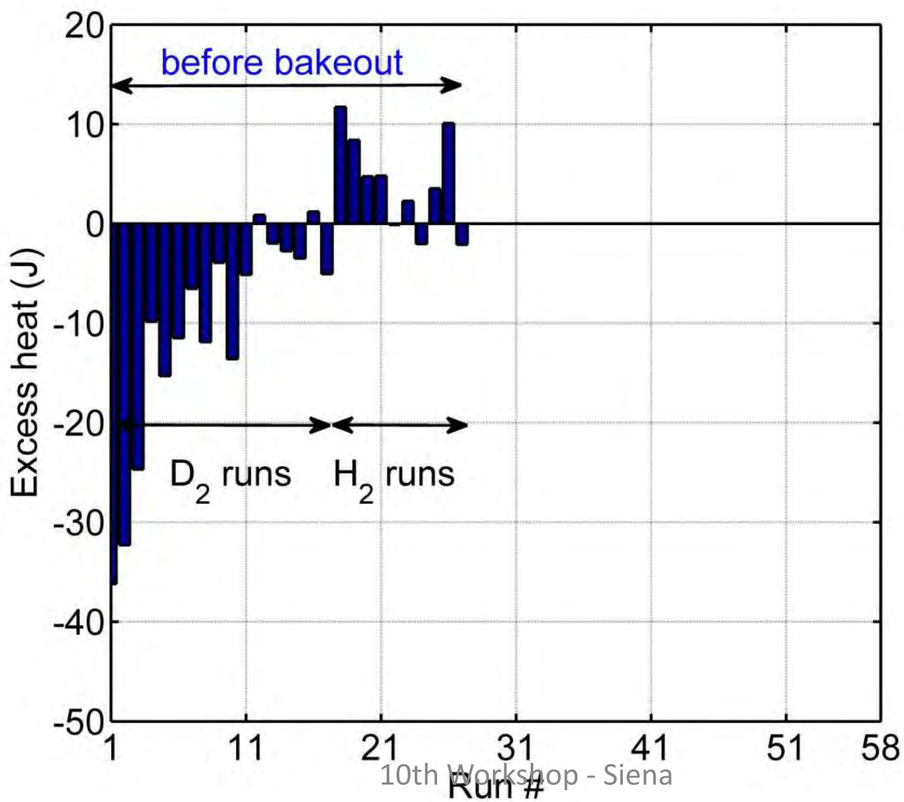
- 8% of mass lost by 400ºC
- Residual gas analysis (RGA): H_2O

Experimental schedule



Experimental schedule

Gas exposure 40ºC → Bakeout 390ºC → Cool down 40ºC → Gas exposure 40ºC



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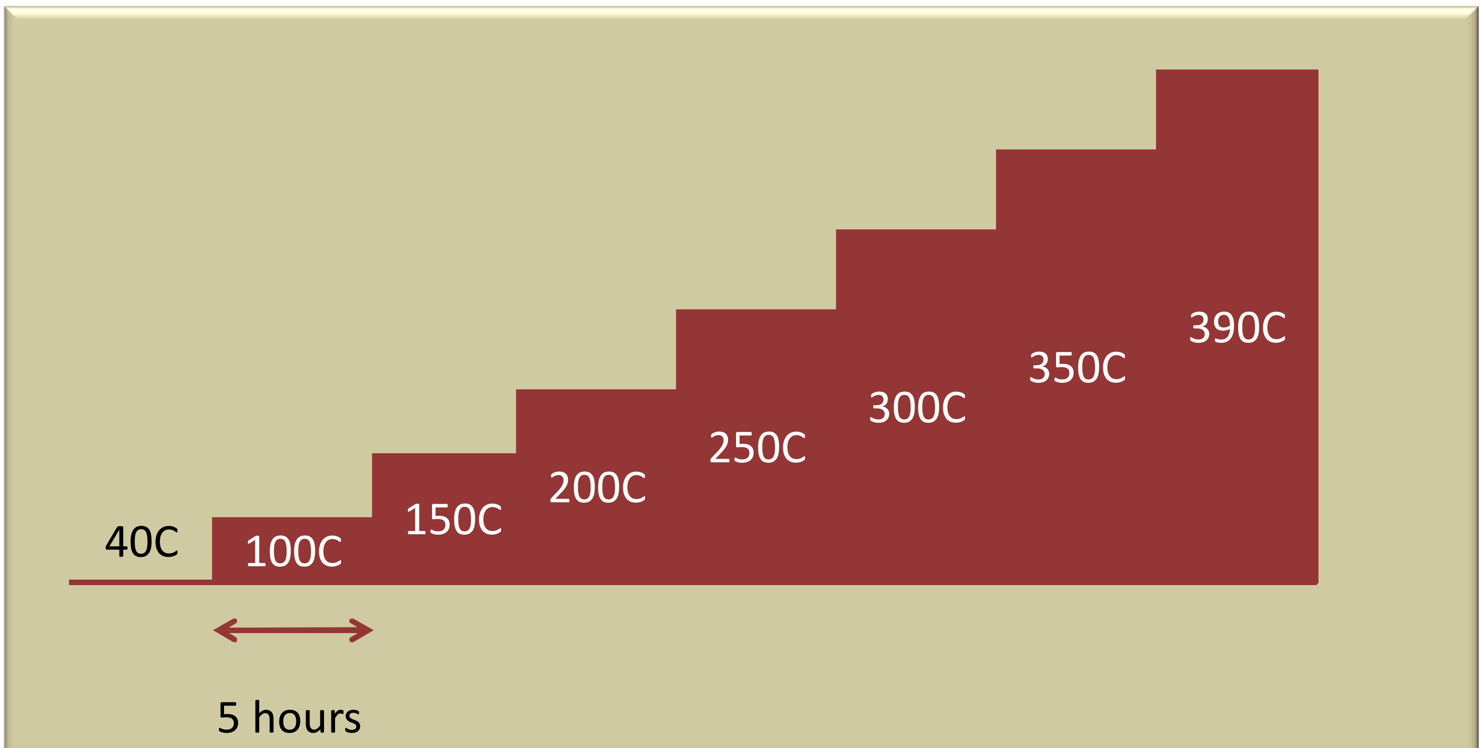
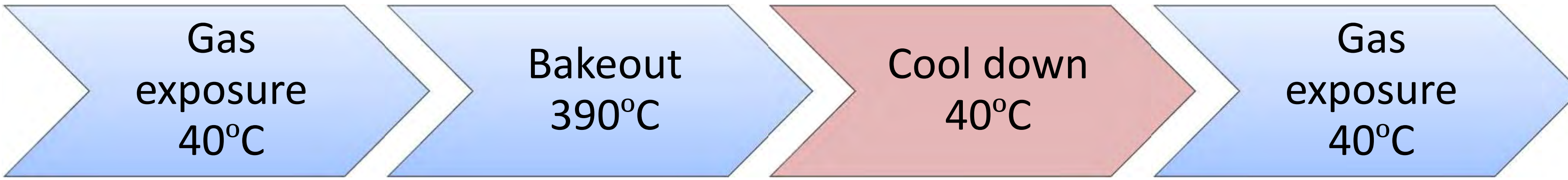
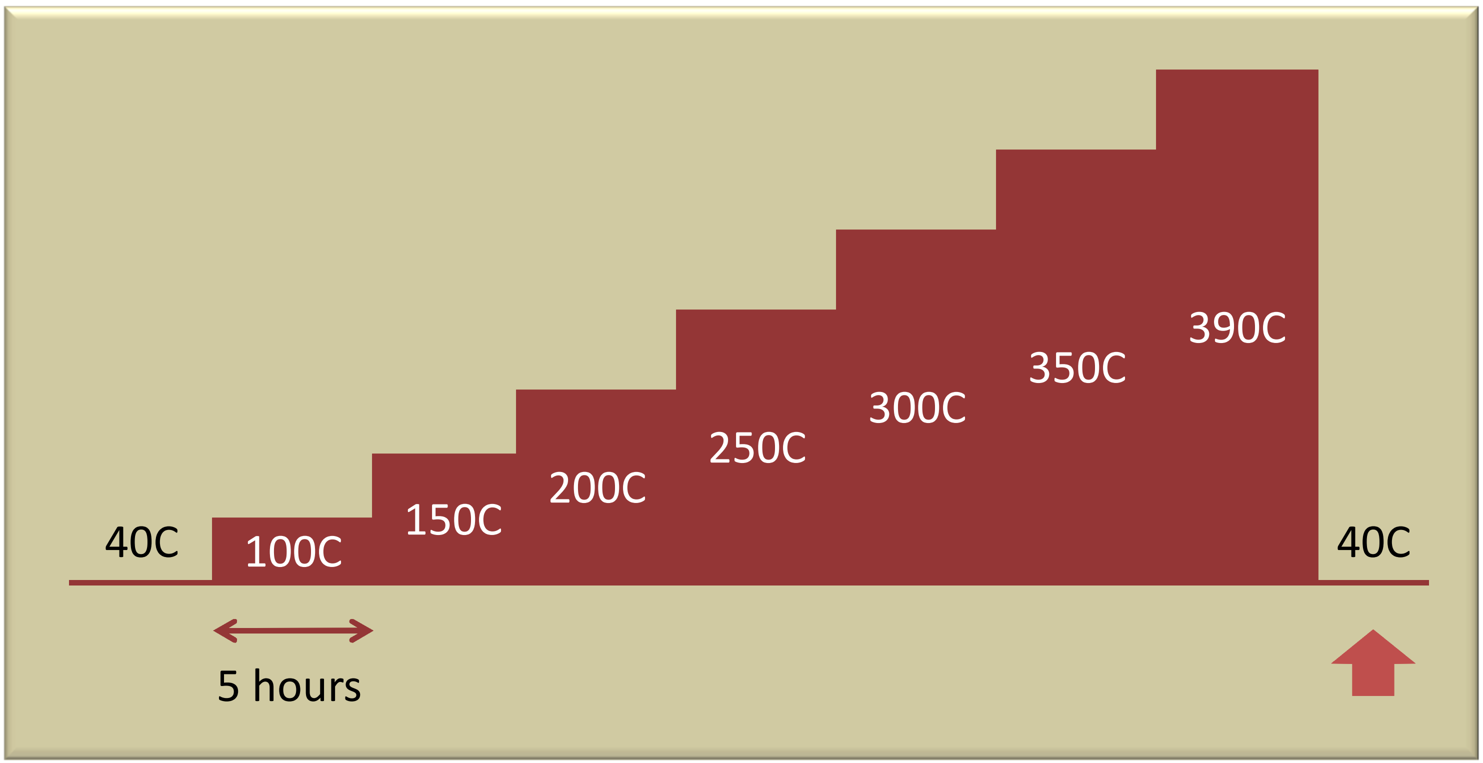


Experimental schedule





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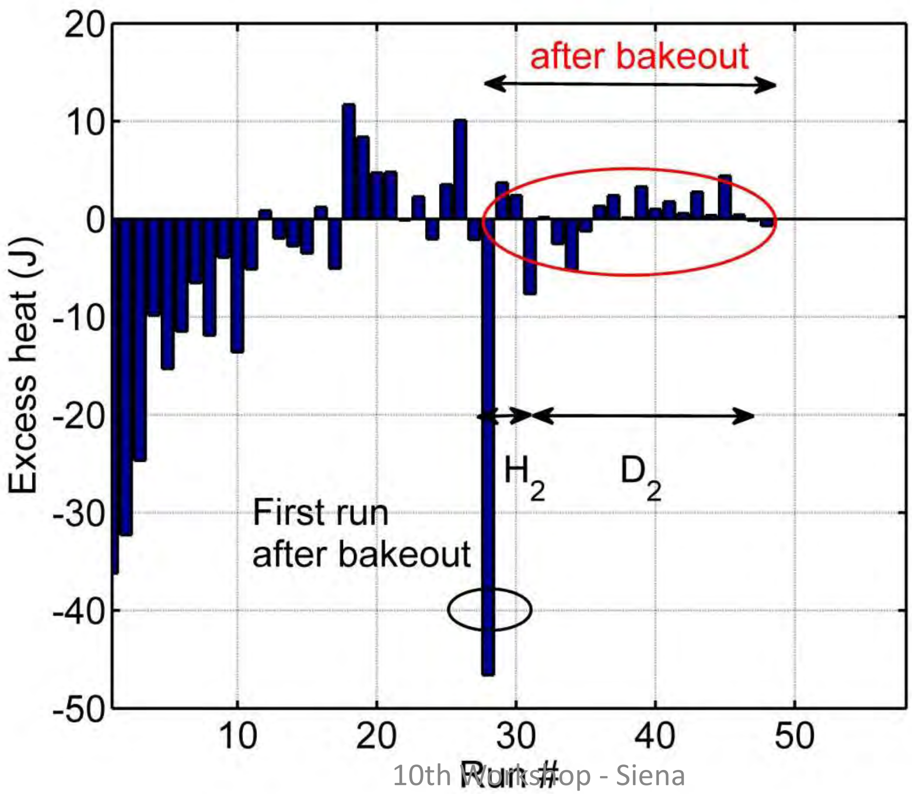
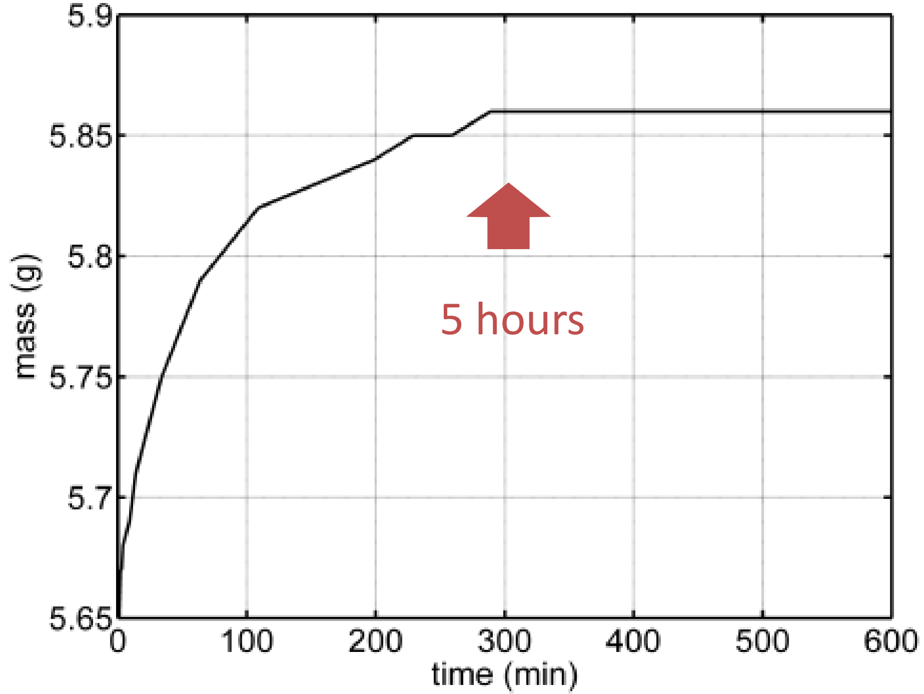


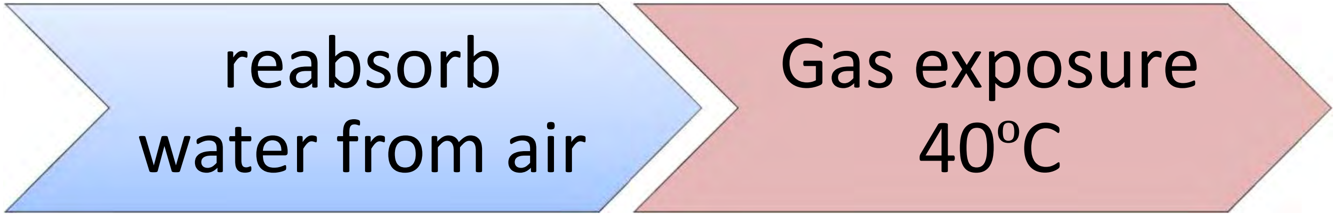
Reactivation of heat production

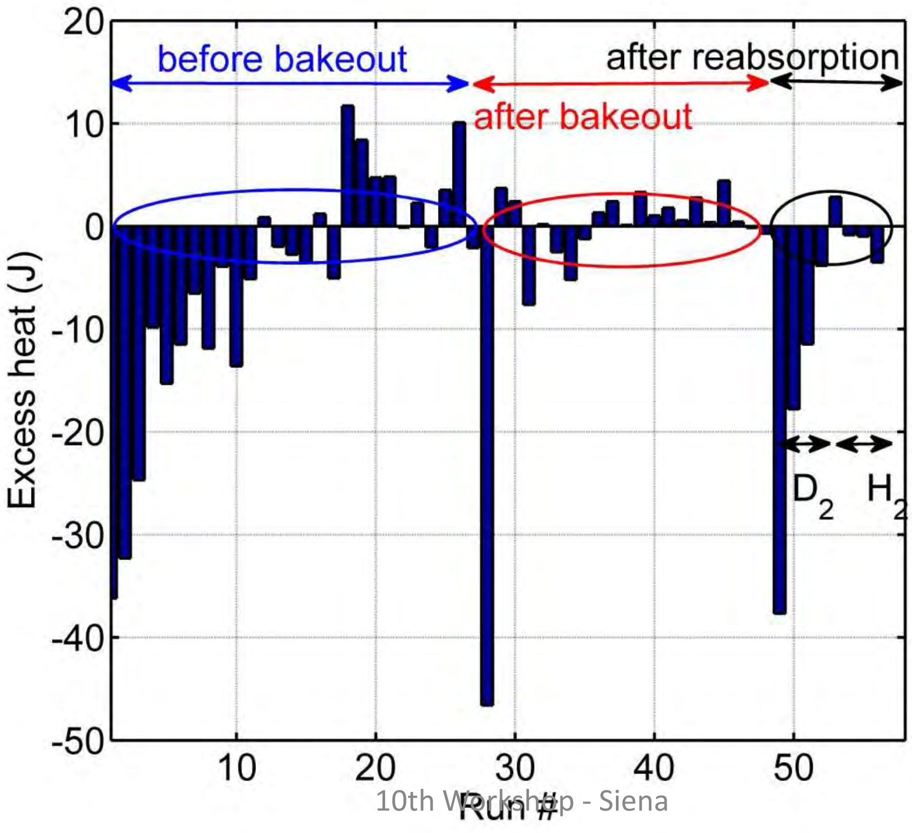
reabsorb water from air → Gas exposure 40°C



- Average weight increase 3.6 – 5%

Reactivation of heat production





Heat available from water

$$H_2O \;\; 5\% \text{ by weight } \Rightarrow 0.3\, g \;\; = 0.017\, mol$$

$$\text{Energy of reaction}: \; -8.3\, kJ \,/\, mol \Rightarrow -141\, J \;\; \text{total}$$

$$\text{Experimentally observed}: \quad -65\, J$$

Summary

- H/D exchange responsible for at least some of excess heat
  - reaction nulled by 390˚C bakeout
  - reaction reactivated by moisture reabsorption

- Calculations show enough water to account for excess heat in our experiment

Conclusion

Baking out the material in vacuum for prolonged time prior to any contact with gases is a way to eliminate potential H/D exchange energy contribution