

**Project title: Creep Damage Assessment Using Hydrogen Desorption**

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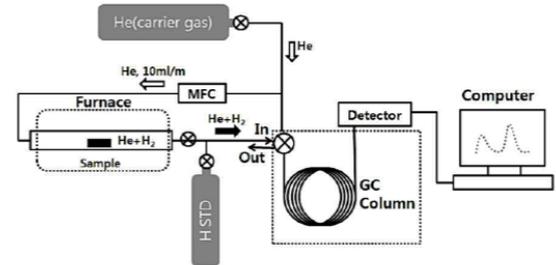
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**Project Highlights:**

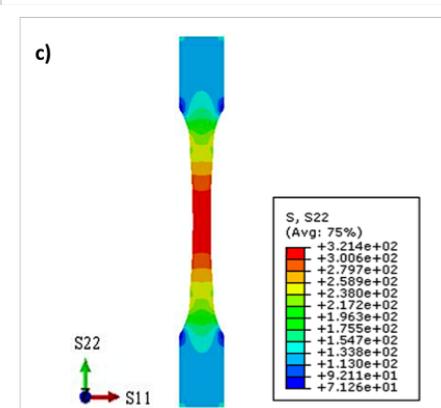
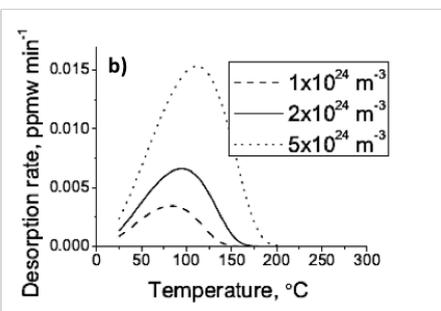
- Modelling and applying hydrogen thermal desorption analysis (TDA) to measure creep damage in austenitic and ferritic materials.
- Cross-validating TDA creep damage models and results by applying other characterisation methods including small angle neutron scattering (SANS), microscopy and serial FIB serial sectioning.
- Contributing to a major programme of research supported by EPSRC, industry and international partners.

**Project Description:**

Hydrogen (H<sub>2</sub>) atoms can diffuse deep into metals causing undesirable embrittlement and material ageing. The ease of absorption depends upon the diffusivity of the material. The reverse process of H<sub>2</sub> desorption is promoted by raising the temperature of the “charged” material. But hydrogen can get trapped in metal by material defects such as dislocations, grain boundaries, vacancies and voids. The desorption rate of hydrogen from a particular kind of trap is a function of the trap binding energy. Thermal Desorption Analysis (TDA) has been developed as a promising method to evaluate the hydrogen detrapping rates for various types of defects, including creep cavities (see Figure 1a). TDA experiments involve first electrochemically charging samples of metal with hydrogen (or soaking in pressurised hydrogen). Then the charged specimen is heated up at a constant rate in a furnace during which the rate of hydrogen release (desorption) measured from different kinds of trap sites varies as the temperature increases. The rate is measured using a gas chromatograph (GC), with Helium acting as a carrier gas. The amount of desorbed hydrogen can be measured, since the gas is separated according to its molecular weight. The TDA curve features can provide information about the binding energy and the number density of trap sites, as shown in Figure 1b. TDA has been developed as promising technique to quantify creep cavitation damage in ferritic and ferritic-martensitic steels [2,3].



a)



c)

**Figure 1:** a) Schematic illustration of the typical set-up for the Thermal Desorption Analysis (TDA) method with gas chromatography (GC), b) the effect of the trap density on the TDA results [1] and c) predicted linear elastic distribution of axial stress across the mid-thickness plane of the stress-range in an hourglass test specimen under uniaxial load.

At the Open University we have successfully developed application of small angle neutron scattering (SANS) to quantify the distributions of carbides and cavities in austenitic stainless steel. We have also applied complementary optical and SEM methods for characterising the microstructure and creep damage assessment. SANS has proven to be an effective

technique for measuring cavities and carbides within the size range of few nm up to 400 nm diameter. However, it is very difficult to get access to research SANS beam time at Central Facilities (and beamtime is prohibitively expensive to buy). The H<sub>2</sub> desorption technique can be carried out in the laboratory and offers the promise of quantifying cavitation damage in volumetric samples of metal at relatively low cost.

The objective of the proposed PhD project is to carry out theoretical and experimental studies exploring the potential for applying TDA to measure creep damage in high temperature materials used in power generating plant. Face centred cubic materials (e.g. pure Nickel and austenitic stainless steel) and potentially body centred cubic ferritic-martensitic steel will be examined.

### Research Methods:

The correlation between TDA curves and creep cavity traps is poorly understood [3]. Therefore, we initially propose studying a reference sample of hiped Nickel powder that has controlled levels of porosity (i.e. known distributions of cavities in the 5 to 400 nm diameter range). Experimental measurements will be coupled with thermodynamic models in order to study TDA in this simple austenitic face centred cubic material. We will determine whether the response can be correlated with population of cavities (size and number density) and assess how the results depend on the material diffusivity and sample size. If the technique looks promising for face centre cubic materials the project will focus on its application to stainless steels. If the technique proves unsuitable for practical measurements, we will direct the project to study ferritic-martensitic steel creep damage (reproducing and extending Japanese work in the field steels [2, 3]).

The project will also deploy novel (hourglass) test specimens in creep tests that give variable creep strain and damage along the length (because the applied stress varies, see Figure 1c). TDA will then be undertaken on samples extracted from different positions along the sample and other characterisation methods (DIC for creep strain, SANS, microscopy and serial FIB serial sectioning) applied to the same samples for validation purposes.

### Indication of project timeline:

**Year 1:** Develop models and carry out TDA measurements on reference samples of hiped Ni powder with controlled porosity (samples from the OU's EPSRC project). Compare results with measurements made using other techniques. Complete Probation Report.

**Year 2:** Develop models and experimental methods for stainless steel (or potentially ferritic-martensitic steel). Set-up validation "hourglass" creep tests.

**Year 3:** Cross-validate TDA models and measurements of creep cavities on hourglass samples with creep damage measured by other techniques (SANS etc.). Complete thesis write-up.

### References

- [1] E.J. Song, Hydrogen desorption in steels, PhD thesis, Pohang University of Science and Technology, 2015.
- [2] S. Komazaki, T. Honda, T. Sakamura, K. Sawada, K. Kimura and Y. Kohno, Change in hydrogen desorption characteristics of Mod. 9Cr-1Mo steel due to creep, J. Iron Steel Inst. Jpn. 96 (2010) 614-619.
- [3] H. Yamashita, S. Komazaki, K. Sato, K. Kimura, Creep damage evaluation of heat resistant ferritic stainless steel by hydrogen thermal desorption analysis, J. Iron Steel Inst. Jpn. 102 (2016) 630-637.
- [4] Y. Zhou, Z. Fan, X. Chen, P. Xu, **H. Jazaeri, P. J. Bouchard**, Theoretical study on the correlation between thermal desorption spectrum features and creep damage, Scripta Materialia, under review.

### Further details:

Students should have knowledge of the physics and thermodynamics of metallic materials, including high temperature creep. The candidate should be self-motivated, show enthusiasm for learning new techniques, have aptitude for experimental work and be able to tackle challenges in a methodical way. Experience in modelling and experimental methods is desirable. The student will join a well-established team researching in the area of High Temperature in the Materials Engineering Group at the Open University.

### Candidate Applications

- 1000 word cover letter outlining how they are equipped in their educational background and expertise to conduct the research project,
- a CV including contact details of two academic references
- An Open University application form, downloadable from: <http://www.open.ac.uk/postgraduate/research-degrees/how-to-apply/mphil-and-phd-application-process> (Note: This is an Advertised studentship and you do not need to submit a proposal).
- IELTS English Language test scores on application. An average of 6.5 and no less than 6 in anyone of the four components. Applicant should have these results when applying.

Applications should be sent to [STEM-EI-Research@open.ac.uk](mailto:STEM-EI-Research@open.ac.uk) by 28 February 2019