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TECHNICAL DESIGN NOTE

# An inert-gas furnace for neutron-scattering measurements of internal stresses in engineering materials

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**Abstract**
The ENGIN-X beamline is a dedicated engineering science facility at ISIS optimized for the measurement of strain, and thus stress, deep within crystalline materials using the atomic lattice planes as an atomic 'strain gauge'. Internal stresses in materials have a considerable effect on material properties including fatigue resistance, fracture toughness and strength. The growing interest in properties of materials at high temperatures may be attributed to the dynamic development in technologies where materials are exposed to high-temperature environment for example in aero-space industry or fission and fusion nuclear reactors. This paper describes in details the design and construction of a furnace for neutron-scattering measurements of internal stress in engineering materials under mechanical load and in elevated temperature environments, designed to permit a range of gases to provide non-oxidizing atmosphere for hot samples.

**Keywords:** neutron diffraction, strain, high temperature, inert gas

Q2 (Some figures may appear in colour only in the online journal)

## Introduction

The ENGIN-X is the dedicated materials engineering neutron beamline at the spallation neutron and muon source ISIS [1]. The ENGIN-X is a world leading neutron diffractometer purpose-built for *in situ* stress evaluation in material samples as well as in engineering components.

The advantage of the diffraction method is the possibility of studying mechanical properties of polycrystalline materials separately in each phase and in groups of grains with a specific orientation. Due to its selectivity, diffraction is a powerful tool used to analyze the mechanical behavior of polycrystalline materials at the mesoscale (phase and/or grain scale).

On ENGIN-X, a range of sample environment equipment, such as *in situ* mountable servo-hydraulic stress rigs, radiant furnace [2] or cryogenic mechanical stress rig [3], is available for a variety of experiments. However, a number of experiments require a radiant furnace integrated into the stress rig with an inert-gas atmosphere sample environment. This sample environment can be effectively used in the investigation of the microstructure evolution behaviors involving phase transformations [4, 5], bulk texture evolution [6], heterogeneous elastic/plastic deformation [7, 8] and dynamic re-crystallization [9, 10] within air or gas atmospheres. At high temperatures, the majority of engineering materials are affected by high oxidation, which affects the material performance. The oxidation layer can be beneficial or detrimental to the material performance. For example, it was suggested by Conrad [11] and Williams *et al* [12] that oxygen will reduce the twinning activity of a titanium Q3

 

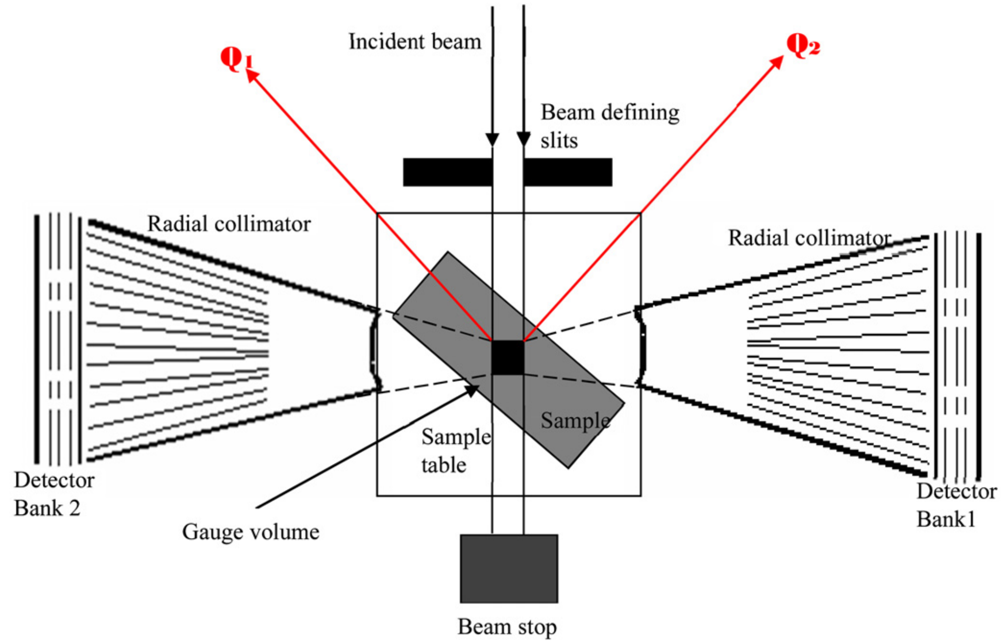


**Figure 1.** ENGIN-X experimental setup for *in situ* strain scanning. (Note that incident slits and collimators provide constrains to the maximum design size of the furnace.) Q4

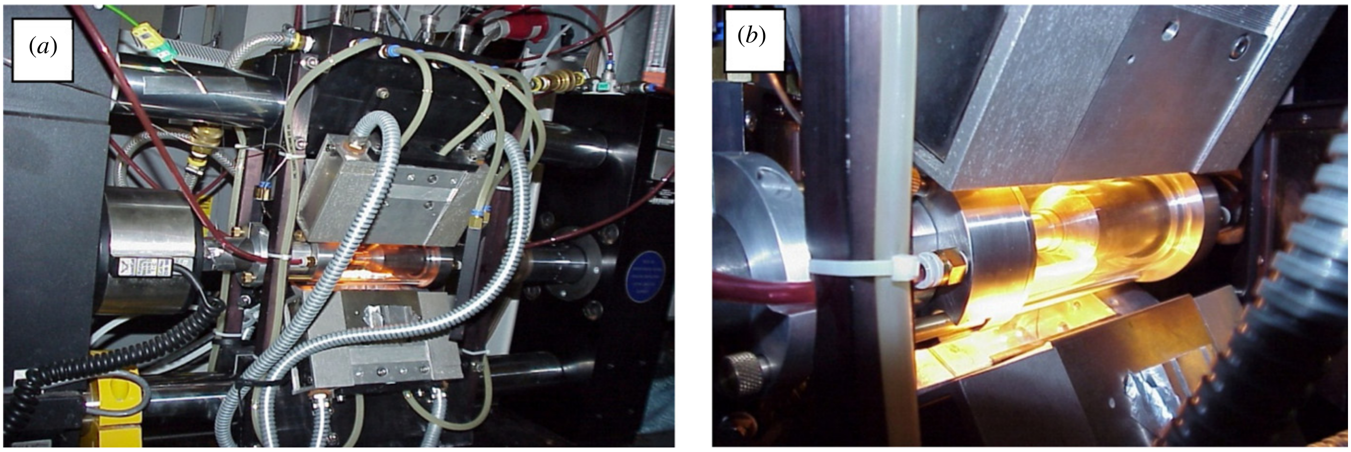


**Figure 2.** Photographs showing the previous method of providing inert atmospheres for the Engin X furnace: (*a*) overview of the setup and (*b*) close-up of the sample area and the glass canister.

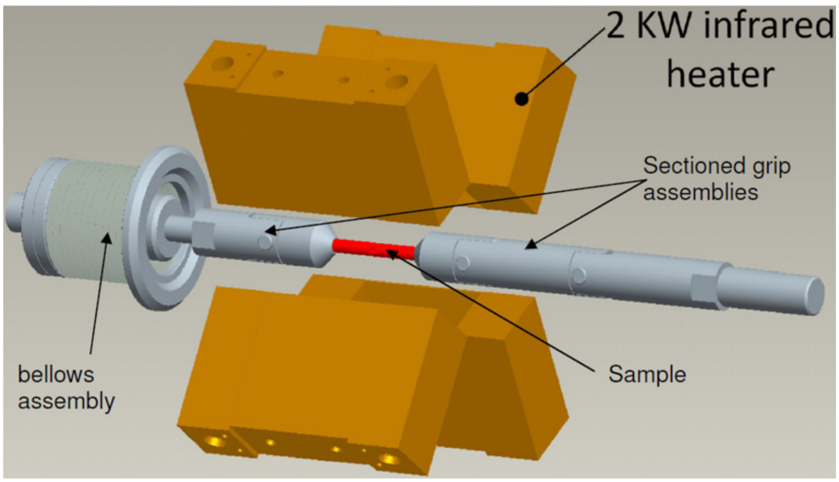


**Figure 3.** The experimental sample mounted between sectioned grip assemblies of stress rig and four 2 kW infrared heaters, positioned such that their focal zones coincide with the sample.

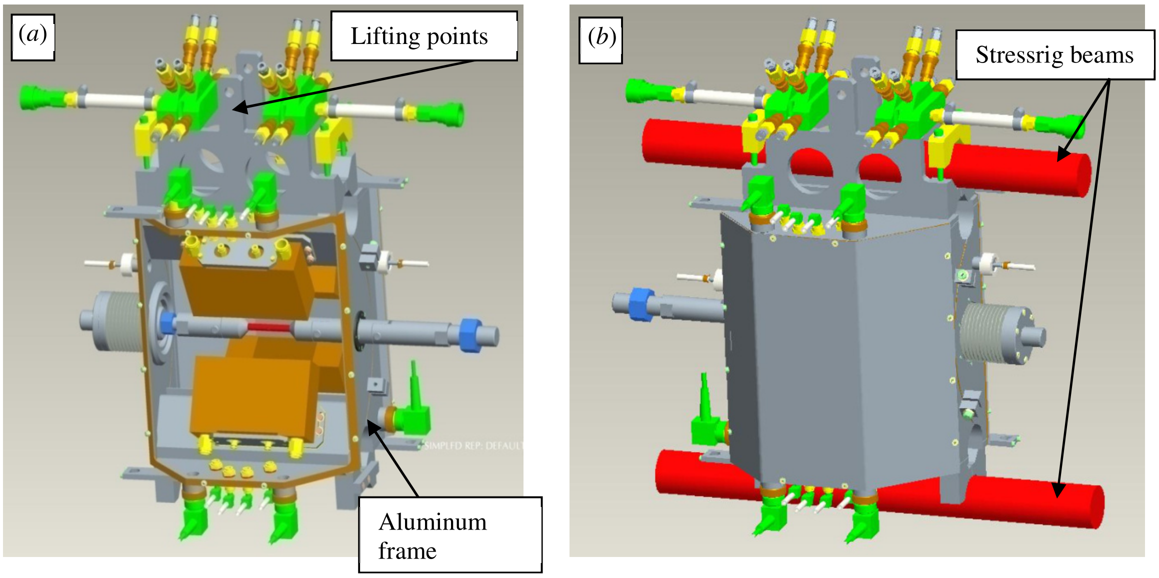


**Figure 4.** The aluminum frame that locates the heaters in position on the stress rig. (*a*) The assembly with all the fittings added for cooling water and power for the heaters, sensors and inert-gas feedthrough and (*b*) the complete assembly attached to the cross-beams of the stress rig (shown in red).

alloys. Another very important benefit of gas furnace is the possibility of investigating hydrogen embrittlement in a range of materials [13]. For instance, the zircaloys in hydrogen environments form hydrides, which is one of the main degradation sources for this group of metals and play a critical role in their failure mechanisms [13, 14]. Currently, the precise mechanism of both oxidation and hydrogen embrittlement is still not well explained. This is mainly due to the lack of the *in situ* experimental characterization of bulk specimens under realistic conditions. These processes are extremely complex and full understanding of the process parameters influencing the materials behavior is vital. The possibility of using the inert-gas furnace *in situ* experiments on ENGIN-X provides an excellent opportunity to investigate these complex phenomena in detail under stable conditions. The furnace integrated into the load frame allows the mechanical testing of high-temperature structural materials, such as metals, superalloys, ceramics and intermetallics, and their composites.

There are several high-temperature sample environment setups described in the literature which are suitable for neutron-diffraction experiments [15, 16]. However, the unique design of ENGIN-X, in combination with a specific rig, requires the development of a novel inert-gas furnace chamber for mounting on the existing ENGIN-X mechanical loading rigs. The design specifications required heating up to 1100 °C for applied loads up to 100 kN, with access for the incident and diffracted neutron beams at 45° to the tensile/compression loading axis.

## Technical aspects

The ENGIN-X instrument is equipped with either a 50 kN or 100 kN stress rig. For *in situ* experiments, the stress rig is mounted on the diffractometer, with its loading axis oriented horizontally at 45° to the incident beam. The two detector

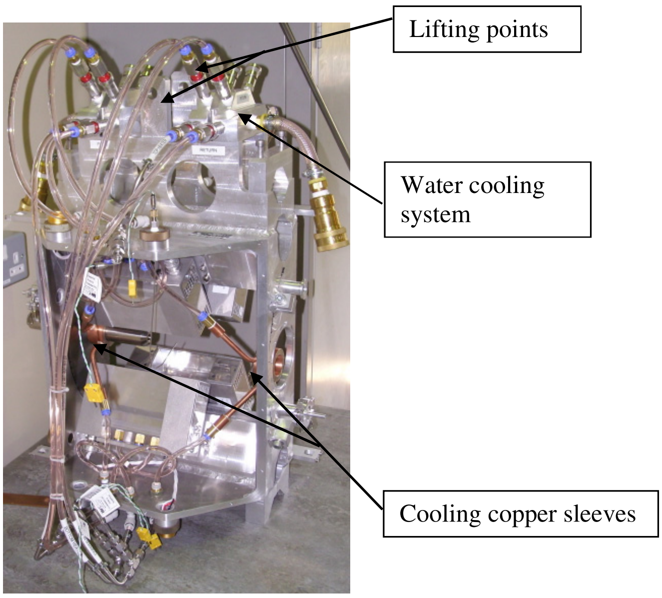


**Figure 5.** ENGIN-X inert-gas furnace off beamline, showing a compact easy-to-access assembly with lifting points cooling system providing cooling water to the lamps and cooling sleeves on the stress-rig grips.

banks allow simultaneous collecting of the time-resolved diffraction patterns at fixed horizontal scattering angles of $\pm 90°$. On ENGIN-X, one can define a small measurement volume (gauge volume) in the sample in the order of a few cubic millimeters. This is achieved by collimating the incident beam (width × height) and by using a radial collimator in front of the detectors to accept only neutrons from a certain depth along the incident beam direction. For the majority of the *in situ* measurements, the largest gauge volume $4 \times 4 \times 4$ mm$^3$ is used. The collimators and the incident slits provide constrains to how big any environment can be positioned on the Engin-X

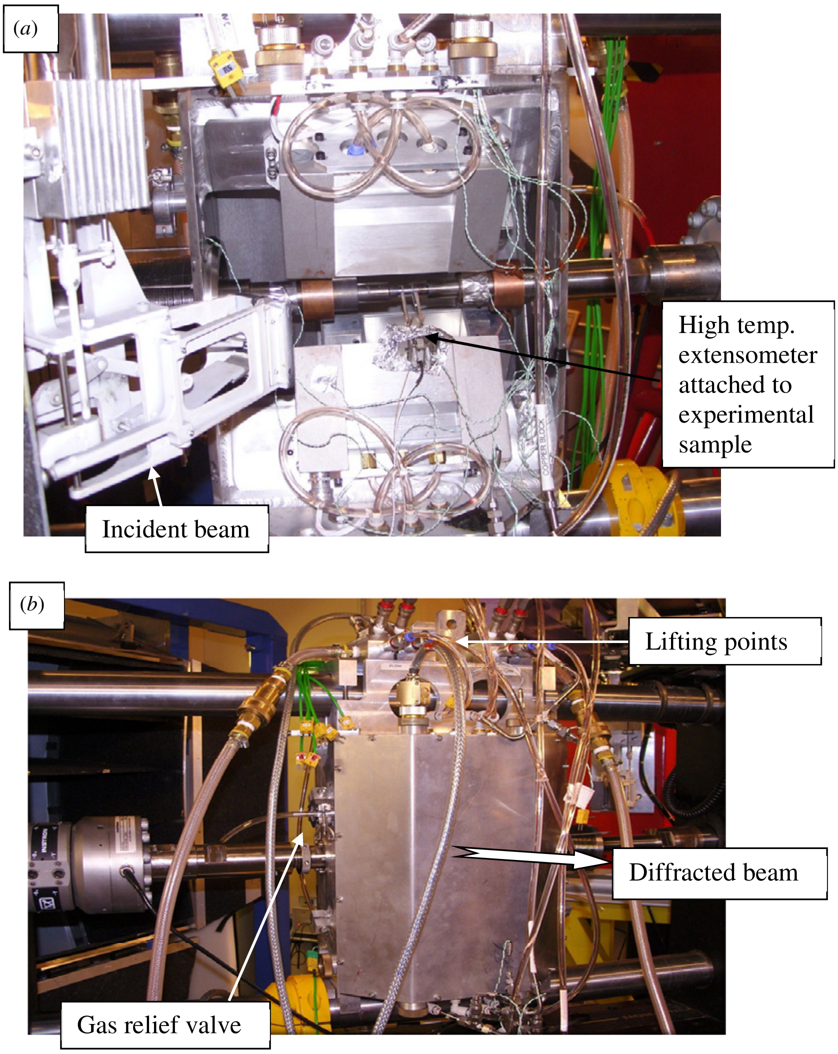


**Figure 6.** Engin-X inert-gas furnace overview of the experimental setup during the experiment: (*a*) incident beam side and (*b*) beam stop side.

sample table. Additionally, the setup and sample changeover should be as quick and easy as possible since the beamline is highly oversubscribed, and consequently, the measurements time is very high in demand.

Previous experiments on ENGIN-X involving neutron-scattering measurements of internal stress in engineering materials under load that required the sample to be in an inert atmosphere were carried out with the sample contained in a glass canister that contained the inert atmosphere. This furnace can be seen in figure 2. Figure 2(*a*) shows two of the four infrared heaters mounted, via mounting brackets, onto the cross beams of the stress rig. The heaters are located such that their focal zones are coincident with the sample. Figure 2(*b*) shows a close up of the sample area and the glass canister. The furnace was originally designed without provision of an inert atmosphere: the glass canister was added with increasing demand for samples to be heated in an inert atmosphere. The experimental setup and sample changing were difficult and time consuming. Due to localized heating of the glass canister, it was prone to cracking. Additionally, it was not possible to attach the high-temperature extensometer for macroscopic measurements, which is often vital for this type of investigation. In order to resolve these problems, a more reliable method of containing the sample in an inert atmosphere during the thermo-mechanical loading experiment on Engin-X needs to be developed.

It was decided to design a large container to house the furnace (the heaters and their mounting brackets) as well as the sample in the inert atmosphere. This would reduce the risk of overheating the container and make the experimental setup more user friendly. The additional demand was that the design needs to be flexible to run in an air environment when required.

### *Furnace design and assembly*

For clarity, figures 3–6 shows models of the furnace being built up around the sample. Figure 3 shows typical tensile sample (in red) mounted between sectioned grip assemblies. The grips are made up of Inconel to maintain their strength at high temperatures. Sectioning the grips allowed easier mounting of the sample within the large container. Note also the bellows assembly to the left of the figure, which allows experiments on samples that will produce large strains in loading without damaging the seal and spoiling the inert-gas environment. Using bellows was an alternative to o-rings sealing around the grip assemblies, which would have been required to be continuous pieces rather than sectioned. Figure 3 shows the addition of four 2 kW infrared heaters, positioned such that their focal zones coincide with the sample. The lamps are positioned to provide a heating window of uniform heating of 20 (width) × 70 (length) mm$^2$. Figure 4 shows the aluminum frame that locates the heaters in position. This frame is made up of two parts fastened together. The gasket material between the two provides a gas seal. Figure 4(*a*) shows the assembly with all the fittings added for cooling water and power for the heaters, sensors and inert-gas feedthrough. Figure 4(*b*) shows the complete assembly attached to the cross beams of the stress rig (shown in red). It also shows a thin aluminum window plate assembled to the frame through which the neutron beam will pass. Again there is gasket material between the plate and the frame.

Note that although there is gasket material to provide good sealing of the furnace to contain the inert gas, the assembly is not classed as a pressure vessel (and indeed there may be small leaks present) and a pressure relief valve (set at 1.3 bar) is attached. Perfect sealing is not required as the aim of the large container is solely to enable air to be flushed out by the inert gas that is gently flowed through the vessel.

*Furnace setup and operation*

The furnace setup consists of two separate parts (figures 4 and 5) for easy assembly on the stress rig. The total weight is ~50 kg. Due to the large weight, a crane system available in ENGIN-X is needed to assemble the furnace on the stress rig. Special lifting brackets are designed for that reason (see figures 4 and 5).

The overview of the inert-gas furnace is shown in figure 6. During the experiment, the temperature sensor is located on the experimental sample, usually attached by spot welding or spring clips for the materials where spot welding can be harmful. This sensor is controlling the heat output of the furnace. The furnace power is supplied by a three-phase solid-state relay (SCR output 200A) and controlled by Eurotherm 2404. The typical furnace output is shown in figure 7. Very good temperature stability was established on the samples within the measurement region for the compression as well as tensile tests. Additional thermocouple sensors are distributed across the chamber to monitor the temperature of the holding grips, the gradient within the chamber and cooling water. During the experiment, only the sample and the grips are getting relatively hot. The grips are designed to withstand high temperature gradients; however, after the cooling sleeves, the temperature is lower than 100 °C. The heating lamps are also cooled by water. For a safety reason, the cooling water system has the temperature sensor on the water exit, and if the water temperature exceeds 35 °C, it trips the system and the furnace cools down. The furnace has been tested to 1100 °C, and all the temperatures were stable. The sensor on the chamber parts did not exceed 35 °C. The cooling sleeves on the grips

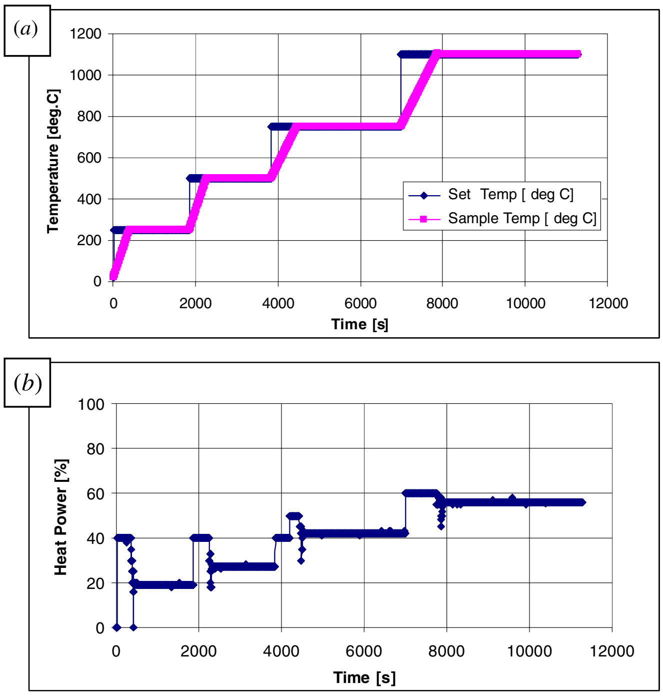


**Figure 7.** Engin-X inert-gas furnace output: (*a*) temperature and (*b*) heat output versus time on the P91 steel (9% Cr, 1% V) tensile specimen.

stabilize the temperature during the experiment as well as allow quick cooling of the sample and jaws to facilitate quick sample changes.

The argon gas of 99.992% purity is usually used but even purer gas source could be implemented. The flow of the inert gas can be varied between 1–10 l min$^{-1}$. Usually, in the first stage of experiments, large flows of 10 l min$^{-1}$ are used to initially fill the chamber quickly, and then, for the duration of the experiment, the flow is set to 3–4 l min$^{-1}$.

## Conclusions and future plans

The new inert-gas furnace environment for the ENGIN-X stress rig has enabled neutron-scattering measurements of bulk stress at temperatures up to 1100 °C and at applied loads up to 100 kN carried out in the non-oxidizing atmosphere for hot samples. The furnace was already successfully used in several experiments [17, 18]. Additionally, this new ENGIN-X inert-gas furnace was adapted to provide not only gas shielding to prevent oxidation but also rapid cooling using argon gas to investigate the deformation-induced ferrite transformation [19, 20] in high-strength low-alloy steels with the high level of detail.

The new furnace can potentially be used across many different disciplines, including physics, materials science, mechanical engineering, aerospace engineering and nuclear engineering. This unique equipment, available now on ENGIN-X, advances the integrated research interactions among the various disciplines and facilitates the development of new interdisciplinary programs combining mechanical behavior of materials at high temperatures with neutron science and engineering.

In future, the setup will be upgraded by the addition of sapphire windows for visual access which will allow a combination of neutron measurements with the image correlation, especially where surface oxidation at high temperature is detrimental to the test and needs to be prevented.

## Acknowledgments

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