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Novel testing chamber for neutron scattering measurements of internal stresses in engineering materials at cryogenic temperatures

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Abstract
The ENGIN-X beam line is a dedicated engineering science facility at ISIS. It is 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 low temperatures may be attributed to the dynamic development in cryogenic technology. This paper will describe a novel cryogenic testing chamber for neutron scattering measurements of internal stress in engineering materials under load at temperatures from 30 K to 300 K.

Keywords: cryogenics, stress measurement, sample environment

(Some figures in this article are in colour only in the electronic version)

Introduction

ENGIN-X is the dedicated material engineering neutron beamline at ISIS [1]. The primary function of the beamline is the determination of residual stresses within the interior of bulk engineering components and test samples, in particular for the development of modern engineering processes (e.g. inertia welding, laser peening) and structural integrity investigations. A second important function of the beamline is for studies of the fundamental material behaviour, such as composite and rock mechanics, the basic deformation mechanisms of metals and phase transformations in shape memory alloys and ferroelectrics. A range of sample environment equipment such as hydraulic dynamic mechanical testing rigs or radiant furnace [2] is available for these investigations.

Until recently, there has been a little capability for studies of *in situ* mechanical behaviour at sub-ambient temperatures, with the few studies which have been performed utilizing a simple cooling mechanism using refrigerated oil, achieving sample cooling to a limit of approximately −20 °C (see, e.g. [3]). There has been a growing demand for investigations of material mechanical behaviour at cryogenic temperatures due to the recent explosive progress in cryogenic technologies. Applications include cryogenic texture processing of zirconium nuclear alloys, strain sensitivity of superconducting magnet wires, cryogenic structural steels and low temperature shape memory alloys for space applications. In addition to these specific applications, the ability to go down in temperature is generally useful for temperature-dependence studies of deformation modes in metals because of the general increase in yield stress with falling temperature. Thus, at lower temperatures, data quality is generally improved because of greater elastic strain partitioning, whereas the rapid fall in yield stress of many materials above room temperature tends to result in lower quality, poorly resolved data.

To address this need, a novel cryogenic testing chamber has been developed for mounting on the existing ENGIN-X mechanical loading rigs. The design specifications required sample cooling down to 70 K or below for applied loads up

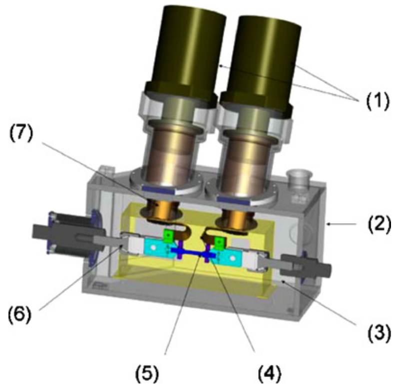


Figure 1. Schematic diagram of the ENGIN-X cryogenic stress rig.

to 50 kN, with access for the incident and diffracted neutron beams at 45° to the tensile loading axis.

Design of the cryogenic stress rig

The cryogenic testing chamber is designed to be cryogen free; instead the cooling power is provided by a closed cycle refrigerator (CCR) [4, 5]. Cryogen-free systems offer the advantage of operational simplicity, generally require much less space and are potentially more 'environmentally friendly' than conventional cryogen-cooled systems.

The cryogenic stress rig is schematically depicted in figure 1. The system consists of: (1) two Leybold 1245 CCRs; (2) an outer vacuum vessel with aluminium windows to provide access for the incident and scattered neutrons; (3) the infrared radiation shield which is connected by thermal links (7) to the first stages of the CCRs; (4) the thermal links connecting the CCR second stages to the rig jaws; (5) the test sample; and (6) the 50 kN hydraulic stress rig.

The hydraulic test rig jaws are by necessity substantial and metallic and thus capable of transferring a large amount of heat to the sample by thermal conduction.

In the cryogenic chamber, heat is intercepted and removed by the CCRs' two cooling stages; first by linking the radiation shield through copper components to the first stages and secondly by linking the sample grips to the CCR second stages through copper shims. These flexible shims allow for movement of the rig during loading and should the sample break during testing.

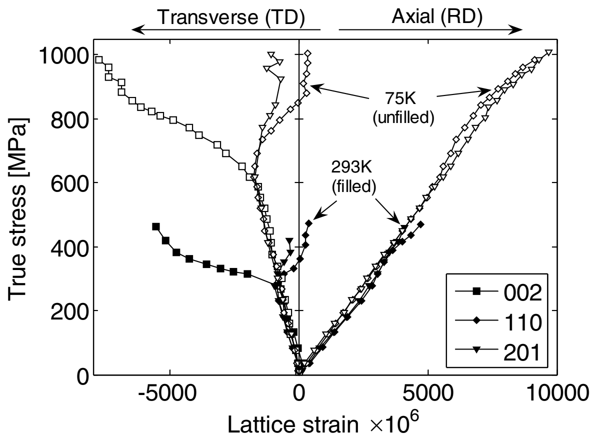


Figure 3. Grain family strain responses of Zr-2 at 293 K and 75 K.

The rig itself incorporates insulating ceramic spacers within the two rig arms which also reduce the heat conducted to the sample. Heat transferred by infrared radiation is intercepted by the shield, and thermal convection effects are minimized by the vacuum in the chamber.

Temperature sensors and heaters are located in clamps attached to each end of the sample holder. The heaters allow warming of the sample and jaws to facilitate quick sample changes.

Figure 2 shows the cryogenic chamber during assembly around the hydraulic test rig on the ENGIN-X beamline.

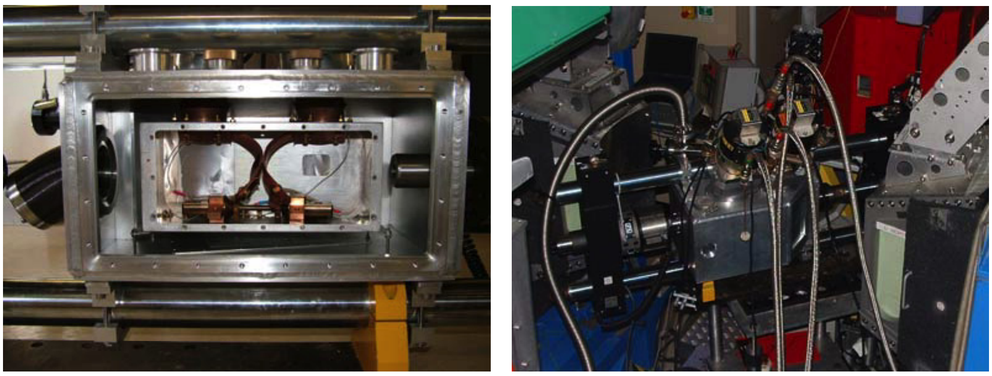
Results

Cryogenic tests

In cryogenic trials, a test sample cooled to a temperature of 30 K in approximately 1 h, more then fulfilling the design specifications of the chamber. A sample change (cold sample to cold sample) was achieved in 2 h. This time should be further reduced with the addition of higher power heaters (currently underway). **Q2**

Preliminary results of neutron diffraction experiment

Figure 3 shows data from the first investigation to benefit: a study of inter-granular stresses in Zircaloy-2 [6]. Subtle



(*a*) (*b*)

Figure 2. The cryogenic test chamber during assembly (*a*) and on the ENGIN-X beamline (*b*).

differences in grain family strain responses between room temperature and 75 K can be interpreted in terms of transitions in activity between the competing slip and twinning deformation modes.

Conclusion

The cryogenic stress rig has enabled neutron scattering measurements of bulk stress at temperatures below 70 K and at applied loads up to 50 kN.

Acknowledgments

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Queries

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