DESIGN AND RATIONALE FOR AN *IN SITU* CRYOGENIC DEFORMATION CAPABILITY AT A NEUTRON SOURCE

V. Livescu[2], T.R. Woodruff[1], B. Clausen[2], T. Sisneros[2], M.A.M. Bourke[2], W.U. Notardonato[3], and R. Vaidyanathan[1]

[1]University of Central Florida
Orlando, Florida, 32816, USA

[2]Los Alamos National Laboratory
Los Alamos, New Mexico, 87545, USA

[3]NASA Kennedy Space Center
Kennedy Space Center, Florida, 32899, USA

ABSTRACT

When performed in conjunction with neutron diffraction, *in situ* loading offers unique insights on microstructural deformation mechanisms. This is by virtue of the penetration and phase sensitivity of neutrons. At Los Alamos National Laboratory room and high temperature (up to 1500°C) polycrystalline constitutive response is modeled using finite element and self-consistent models. The models are compared to neutron diffraction measurements. In doing so the implications of slip and creep to microstructural response have been explored. Recently we have been considering low temperature phenomena. This includes changes in deformation mechanisms such as the increased predilection for twinning over slip. Since this is associated with measurable texture changes as well as microstructural strain effects, it is well suited for study using neutron diffraction. This paper outlines the design and rationale for a cryogenic loading capability that will be used on the Spectrometer for MAterials Research at Temperature and Stress (SMARTS) at the Los Alamos Neutron Science Center (LANSCE).

INTRODUCTION

Low temperature mechanical performance can differ considerably from room or high temperature. Moreover phenomena such as twinning, phase transformations or residual stress can significantly affect mechanical response. Accordingly an understanding of mechanical properties of polycrystalline structural materials at low temperatures is of

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importance to many scientific and military endeavors. Although cryogenic mechanical property data is often, although by no means always available, the fundamental mechanisms associated with deformation at low temperatures remain of interest. Practical examples of low temperature applications include sensors for astronomical observations, semiconductor-based devices, aerospace latch and release mechanisms, thermal switches, valves, and cryogenic storage systems.

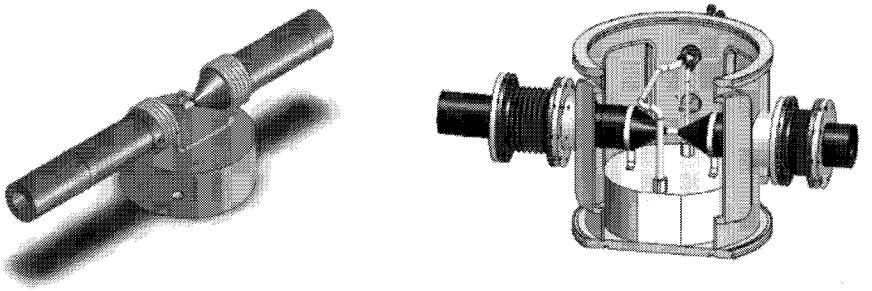
At Los Alamos National Laboratory there is a comprehensive effort to develop models that describe the effects of strain rate, texture and temperature on polycrystalline constitutive performance. To date the focus has been on room and high temperature performance. However recently we have been considering strain and texture effects associated with uniaxial deformation of hexagonal close packed (hcp) metals such as beryllium, magnesium and zirconium [1-3]. More so than cubic materials, the effects of cryogenic deformation are of fundamental interest because of the increased propensity for twinning at low temperature (or high strain rate) in lower symmetry metals. For zirconium in particular twin volume fractions were measured using neutron diffraction for samples that were previously deformed at high strain rates and cryogenic conditions including 77K [4, 5]. Interest in this type of problem has lead us to develop a cryogenic loading capability for *in situ* loading with simultaneous neutron diffraction. This will allow the study of phenomena as they occur and will be a significant improvement over interrupted tests. At the time of writing the capability has been designed and fabrication is underway, preliminary measurements will be made in January 2004.

NEUTRON DIFFRACTION

In engineering calculations, the mechanical performance of structures or components is often calculated under the assumption that the material is homogeneous. Although this assumption is often sufficient, it may become dramatically invalid under atypical temperature or pressure conditions or in the case of directional asymmetries associated with texture effects. The individual grains that comprise polycrystals typically exhibit directionally elastic and plastic characteristics. Neutron diffraction provides a technique to study their influence on macroscopic response. Because neutron measurements can irradiate bulk volumes of materials (as much as several cm^3), the results are not surface limited, which is the case using laboratory x-ray sources. Moreover, the neutrons' weak interaction means that they easily pass through heat shields or vacuum containment simplifying *in situ* deformation studies. Typically the measurements entail the measurement of diffraction spectra from which shifts in the planes position and intensity of lattice planes to applied loads can be measured. The results are used to test the validity of modeling assumptions concerning grain reorientation (i.e., texture development) and the hardening evolution in individual grains. Measurements include both spatially resolved residual stress determinations and *in situ* deformation studies [6].

THERMAL CALCULATIONS

Room temperature uniaxial compression tests in the neutron beam are performed routinely and obviously don't entail temperature variations across the sample. For measurements at high temperature, temperature variations across the sample are possible and must be minimized to avoid experimental ambiguity. Thus before implementing a cryogenic capability we performed a series of calculations to ensure that a uniform



(a) (b)

FIGURE 1. (a) CAD model used for thermal analysis (showing cold mass and copper tubing providing thermal contact with the platens), (b) CAD image showing the engineering implementation of the cooling design (showing vacuum chamber, cold mass and braids providing thermal contact with the platens).

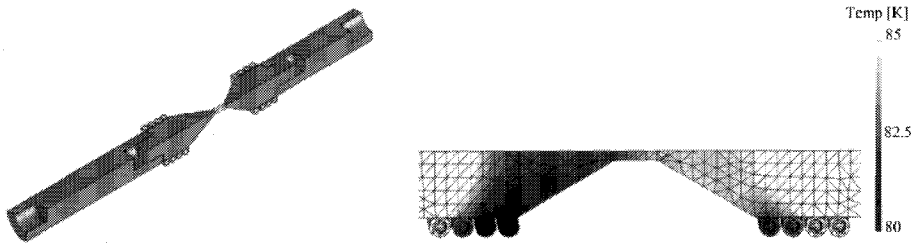
temperature profile across a 24mm long 10mm diameter cylindrical compression sample (typical of a neutron diffraction measurement) was achievable. The heat transfer analysis was performed for the design described below.

The concept comprised a copper cold mass heat exchanger made of 50Kg block of oxygen free copper cooled by liquid nitrogen, which passed through a labyrinth of internal channels. The cold mass is then connected to the platens by copper tubing and enclosed in a vacuum chamber (FIGURE 1). A simplified solid model was constructed in SolidWorks® (FIGURE 2a). Using COSMOS/Works™ design analysis software, the model was meshed and appropriate properties were assigned to the components. The properties and function of the materials used in the model are listed in TABLE 1.

Boundary conditions applied to the model were axial symmetry, application of a zero flux boundary condition to the sectioned surfaces. The temperature of the surfaces in contact with liquid nitrogen is 77K. Natural convection is taken into account across the internal surfaces of the heat exchanger ($h=500W/Km^2$), the external surface of the platens outside the vacuum chamber ($h=3W/Km^2$) and, at a very low level on all the assembly surfaces inside the vacuum chamber ($h=0.1W/Km^2$). These numbers were selected after matching a preliminary model with available data from a fixture attached to the cold mass. The heat transfer inside the vacuum chamber occurs by radiation from the surrounding surfaces with an emissivity coefficient of 0.3 and a radiation shape factor of 0.4. The ends of the push rods lie outside the vacuum chamber. Asymmetric thermal boundary conditions were applied to either end of the model – reflecting the reality of the experimental set-up.

TABLE 1. Mechanical and thermal properties used for the thermal analysis.

Material	E [GPa]	μ	G [GPa]	ρ [kg/m^3]	UTS [MPa]	YS [MPa]	Thermal Cond. [W/mK]	Heat Capacity [J/kgK]	CTE [10-6K^{-1}]
UNS C10200 [*Cold mass*]	117	0.31	44.7	8900	215	49	391	385	16.9
VascoMax C-350 [*Platens*]	200	0.33	75.2	8080	2359	2320	25.26	452.3	11.3
NiTi [*Sample*]	28	0.3	11	6450	754	100	10	320	12.7



(a) (b)

FIGURE 2. (a) Simplified model used for the thermal analysis, **(b)** Steady state temperature profile.

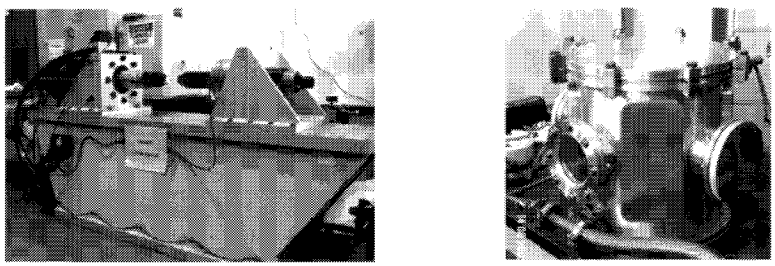
The end outside the chamber of one push rod (which is in contact with the actuator) is at 340K, while the other end, which is connected to the load cell, is at 295K.

The results for the thermal calculation are shown in FIGURE 2b. We conclude that the proposed cooling solution allows for sample steady state temperatures around 82K. The temperature along the sample reached steady state in approximately 1 hour. There is a temperature gradient of about 1.7K across the length of the sample. This is acceptable for the proposed measurements.

ENGINEERING IMPLEMENTATION

The cryogenic testing equipment described below will be used on SMARTS (Spectrometer for Materials Research at Temperature and Stress) for neutron diffraction measurements on engineering materials. SMARTS is a third-generation neutron spectrometer designed for structural materials studies at the spallation neutron source Los Alamos Neutron Science Center (LANSCE) [7]. Room temperature *in situ* compression measurements on SMARTS are performed using a load frame with a capacity of 250KN (FIGURE 3a). The load is applied by two cylindrical compression platens driven by an electro-hydraulic actuator.

For thermal insulation a vacuum chamber was used to isolate the sample from the environment (FIGURE 3b shown without platens inserted). In the neutron diffraction measurements, the chamber must allow the passage of the incident, transmitted and



(a) (b)

FIGURE 3. (a) SMARTS load frame configured for room temperature compression, **(b)** vacuum chamber used in conjunction with platens for cryogenic measurements.

diffracted beams. Since there are detectors on either side of the sample this necessitated four rectangular aluminum windows mounted at 90° to one another.

Flanges, positioned on the same diameter at 45° with respect to the aluminum windows, accommodate the insertion of the two compression platens by means of edge-welded bellows. There is also a clear window viewport. During measurement the chamber is evacuated to approximately 10^{-3}Torr and then a turbo pump is used to further increase the vacuum level. For safety, a pressure-relief valve is included in the chamber to prevent over pressurization in the event of a leak of nitrogen from the cold mass inside the chamber.

The compression platens were designed to accommodate a cylindrical sample 10mm in diameter and 24mm long. Each platen is made of two parts: a tapered tip that allows for sample positioning and alignment and a push rod that makes the connection with the load cell in one side and the actuator in the other side. In choosing the material appropriate for the platens, two characteristics were considered: the compressive yield strength and mechanical properties at low temperatures. Since measurements on bulk metallic glasses or shape memory NiTi specimens may call for applied stresses of -2000MPa or more, the material options for the platens were limited.

In practice we struggled to find a comprehensive suite of mechanical low temperature material properties and chose VascoMax C-350 (a cobalt strengthened, maraging steel with 18% Ni) because of its impressive compressive yield stress of –2675MPa. Other materials that were considered and rejected included: Stellite 6B, a Cobalt base wrought superalloy with compressive strength of 2392MPa, and Austempered Ductile Iron Grade 5 with ultimate compressive strength of 2137MPa.

The cooling system is built on the principle of liquid cooled cold plates. Liquid nitrogen is routed through an internal labyrinth in the solid heat conductive mass that serves as a heat sink. The cold mass is a 10cm thick disk made of oxygen-free high conductivity Copper (UNS C10200), positioned on the bottom of the vacuum chamber. Although the calculations above used copper tubing we anticipate using copper braids as a flexible thermal conduction path from the tips of the compression platens to the cold mass. Is important that the sample is in the neutron beam, which is why there is no direct contact between the cold mass and the sample. The system uses valves and fittings to connect the cold mass to a dewar containing refrigerant (typically liquid nitrogen). To provide thermal control the flow rate of nitrogen through the cold mass will be controlled and electric heaters will be added to the braids connecting the cold mass to the sample. Aluminized Mylar shields are used to minimize the heat transferred to the cold parts by radiation.

At the time of writing the chamber, cold mass and platens have all been procured and

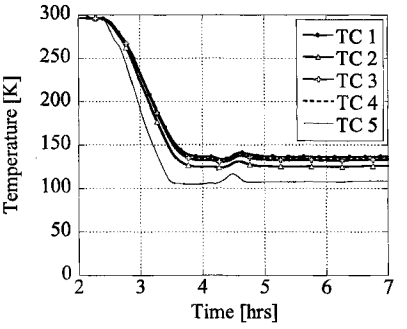


FIGURE 4. Temporal evolution of the cold mass temperature (TC 5) during the experiment.

preliminary cooling measurements showed that the cold mass can be cooled to an average temperature of 105K in approximately one hour (FIGURE 4). This agrees well with the cooling time estimated in the thermal analysis although it should be noted that the 105K steady-state temperature of the cold mass reflects poor insulation. It is hoped that an improved vacuum and better thermal insulation inside the chamber will substantially improve the cold mass minimum temperature. These results were obtained in a poor vacuum (only 10% atmospheric due to a leak from the cold mass into the chamber). The liquid nitrogen usage (with manual control of the flow rate) holding the cold mass at 105K was approximately 160 liters in 6 hours. This experimental set-up was used for spatially resolved measurements on welded coupons from the space shuttle flowliner [8].

PROPOSED MEASUREMENTS

Shape memory alloys (SMA) are of growing technological importance. SMAs are a unique class of alloys that "remember" and return to their original shape due to a thermally induced phase transformation, following deformation. The strain recovery can occur against large forces, resulting in their use as actuators. To date there has been a considerable effort using neutron diffraction to study room temperature transformation SMA phenomena, starting with reference [9]. While SMAs have been commercially used at cryogenic temperatures in single-use applications (e.g., couplings), their use at cryogenic temperatures in cyclic, switch-type applications has been limited by a lack of understanding of the underlying deformation mechanisms (i.e., phase transformation and twinning) at cryogenic temperatures. One application as a thermal switch is outlined in this publication [10].

In situ neutron diffraction experiments on NiTiFe shape-memory alloys during selected combinations of external loading and cryogenic cooling will be performed at Los Alamos National Laboratory. After pre-straining NiTiFe, the phase transformation will be thermally induced by increasing the temperature while exerting a stress on the shape-memory alloy. In this constrained recovery situation, neutron diffraction spectra will be recorded. This testing represents a situation that is identical to a shape-memory alloy working in a cryogenic actuator application such as a thermal switch for cryogenic liquefaction and storage systems, cryogenic valve, seal or self-healing gasket. The aforementioned experiments will result in quantitative, *in situ* measurements of evolving internal strains, texture, phase and twin volume fractions in the cubic, rhombohedral and monoclinic phases of NiTiFe at cryogenic temperatures.

Another series of measurements we propose to make is to study *in situ* twinning that takes place in Zirconium to complement previous work from interrupted tests [4]. Other materials that are likely candidates for study are Al-Li alloys due to their great potential for use in the aerospace industry [11, 12]. They show improvement of their strength, ductility and toughness at low temperatures but the origins of their behavior or deformation mechanisms at cryogenic temperatures are not clear. Microcomposite Cu-Nb conductors manifest a variation in resistivity at low temperatures that could be influenced by features of the nanocomposite structure such as nanoscaled dimensions of the components and high residual stresses [13].

CONCLUSIONS

A design for low temperature loading capability for *in situ* neutron diffraction measurements has been analyzed and implemented. Thermal calculations indicate that a compression specimen can be cooled to a temperature of 82K ± 1K by cooling the tips of the platens adjacent to either end of the specimen.

Preliminary tests using a vacuum chamber and a low temperature cold mass seem to validate the approach although the cold mass reached only 105K on the first attempt. We believe that this is because of a poor vacuum in the chamber. However, the cooling time of approximately 1 hour is consistent with the duration of a series of neutron diffraction tests which typically last 24 hours. Moreover, the amount of liquid nitrogen of approximately 160 liters necessary to maintain the temperature of the cold mass is acceptable.

The first measurement using the new capability will be performed in January 2004.

ACKNOWLEDGEMENTS

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