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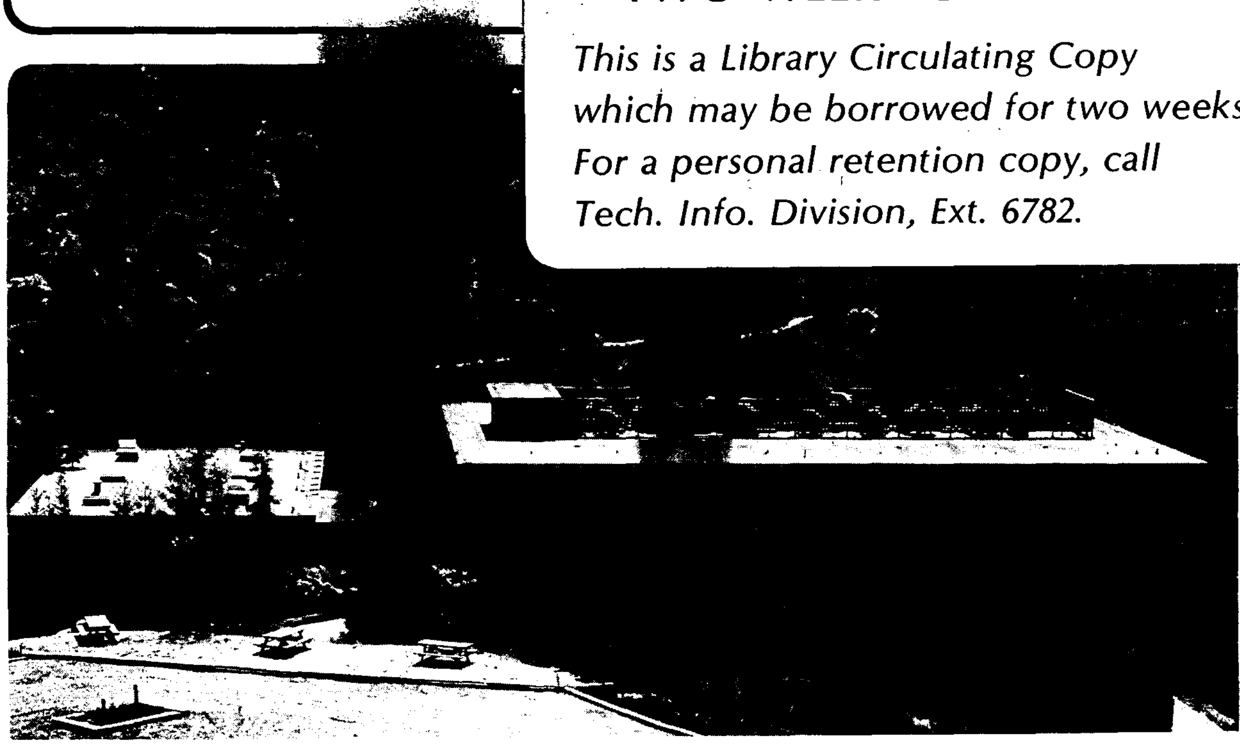

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Abstract

Many important cryogenic structural steels include an austenite phase which is metastable at low temperatures. Combined effects of magnetic fields and mechanical loading on the martensite transformation are briefly reviewed, and implications for the mechanical properties of these steels are suggested. Preliminary experimental data on effects of pulsed magnetic fields on martensite transformations and mechanical properties of 9Ni steel and 304 stainless steel are reported. A new testing facility for measuring mechanical properties at 4K in steady magnetic fields of over 8T is described.

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Introduction

Most existing or planned high field superconducting magnets use austenitic stainless steels for the magnet structure because these materials are available, paramagnetic, and tough at 4K. Many common austenitic (γ-phase) stainless steels are, however, structurally metastable at 4K. They prefer the bcc (α-phase), and will undergo spontaneous martensite transformations under appropriate conditions. Metastable austenite also appears in other classes of cryogenic structural steel. Some of the promising new high-Mn steels are metastable with respect to the indirect transformation $\gamma \rightarrow \varepsilon \rightarrow \alpha$, where ε-martensite is a hexagonal phase. Ferritic steels are commonly toughened for low temperature service by the precipitation of metastable austenite within the ferritic matrix. The martensite transformation is promoted both by high magnetic fields [1] and mechanical loads [2] in ways which are imperfectly understood, but which may affect structural performance or reliability in the high stress, high field regions of the large and powerful superconducting magnets now being developed. This paper briefly reviews the combined effects of magnetic fields and mechanical loading on this transformation, reports two initial experiments with a pulsed magnetic field, and describes a new testing facility which has been established to measure cryogenic mechanical properties in steady 8T magnetic fields.

Effects of Magnetic Fields on Martensite Transformations

A magnetic field is expected to influence a phase transformation whenever the thermodynamic end states of the transformation have different magnetic properties. This is the case when a paramagnetic phase (γ or ε) transforms to the ferromagnetic phase (α). The precise effect of the magnetic field is different for each of the two distinct types of martensite transformation: 1) the athermal transformation, in which the volume fraction of martensite is fixed by the temperature to which the material is cooled, and 2) the isothermal transformation, in which the volume fraction of martensite is a function of both temperature and holding time.

In an athermal transformation, which occurs in high Ni austenites, for example, the imposition of a magnetic field raises the temperature, M_s, at which martensite is first observed, and increases the total fraction of martensite formed. The increase in M_s is expected for the following thermodynamic reason [1]. Assuming that any nucleation barriers remain constant, the equilibrium of a system with given T, P, and applied magnetic field, H, is governed by the modified thermodynamic potential:

$$\Phi = G - MH \tag{1}$$

where G is the Gibbs free energy and M is the magnetic moment (both per unit volume). Two phases (γ,α) are in equilibrium when they have equal values of Φ. A change in H at constant P will hence cause the γ-α equilibrium temperature to vary in accordance with the relation:

$$dT/dH = M_\alpha/\Delta S(T) \tag{2}$$

Fig.1 Enhancement of Athermal Martensite in a Magnetic Field (after [1])

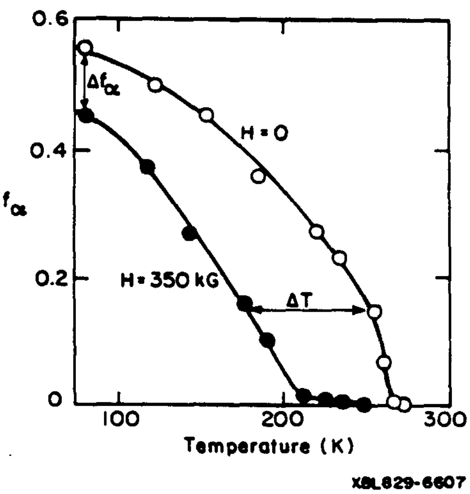
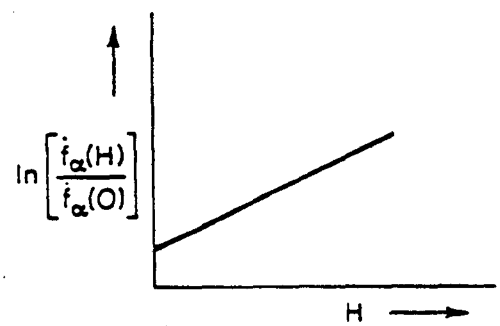


Fig.2 Enhancement of Isothermal Martensite in a Magnetic Field



where M_α is neglected and the entropy change is $\Delta S(T) = S_\gamma - S_\alpha$. The derivative may be nearly 2K/tesla , and is expected to cause a corresponding change in M_s. The increase in the total fraction of martensite seen in Fig. 1 may be due in part to the same thermodynamic effect.

Isothermal transformations are governed by the kinetics of nucleation and growth, which may also be influenced by a magnetic field (see Fig. 2). The behavior is simplest at the beginning of the transformation when the rate of transformation, f_α , should be simply proportional to the martensite nucleation rate:

$$f_\alpha = A \exp[-W/kT] \tag{3}$$

where W is the activation energy for nucleation. The available experimental data [3] suggest that W is linear in ΔG, and hence will be linear in $\Delta\Phi$ in the presence of a magnetic field. The initial transformation rate should then be expressible as:

$$f_\alpha(H,T) = f_\alpha(0,T) \exp[K_2 MH/kT] \tag{4}$$

where K_2 is a constant. The predicted exponential dependence of the initial transformation rate on the magnetic field has been observed experimentally [4] , and may cause an apparently sudden intrusion of the martensite transformation when a high magnetic field is applied [5].

Effect of Mechanical Stress in Magnetic Fields

Mechanical stress also promotes the martensite transformation, since it couples to the shape change associated with the transformation to accomplish work, and thereby lowers the free energy of the martensite phase. In marginally stable alloys the transformation may intrude at stresses below the yield strength ("stress-induced martensite"), but the transformation more often appears after some plastic strain ("strain-induced martensite") at temperatures below the "deformation-onset temperature", M_d. Temperatures of cryogenic service are frequently below M_d, and the present two experiments studied mechanical properties below M_d. Mechanisms that mechanically induce the martensite transformation are expected to operate in the presence of magnetic fields, and thermodynamic considerations suggest that magnetic and mechanical effects on the transformation will be at least additive. We suggest that synergetic effects of combined magnetic and mechanical stresses may also be observed in the mechanical properties of materials with metastable austenite.

The presence of a magnetic field in addition to mechanical loading should influence the mechanical properties of cryogenic structural alloys in at least four situations: 1) when the yield strength of an austenitic steel is controlled by stress-induced martensite, the presence of a high magnetic field should cause a decrease in yield strength. 2) When an austenitic steel undergoes a strain-induced martensite transformation, the presence of a magnetic field should cause an increase in the volume fraction of transformation as a function of stress, and may thereby affect the mechanism of plastic deformation of the material. 3) When an alloy transforms on straining, the presence of a magnetic field should affect those mechanical properties which involve large plastic strains, fracture and fatigue crack propagation, for example. 4) When the material is a ferritic alloy whose toughness at low temperature is achieved through precipitation of a dispersed austenite phase, the presence of a magnetic field may lead to a deterioration of toughness through premature transformation of the austenite.

While none of the phenomena cited above have, to our knowledge, been studied in detail, the second and fourth have recently been observed in simple experiments conducted with a pulsed magnet in our laboratory. The detailed characterization of these phenomena will, however, require an experimental facility capable of imposing controlled and measurable magnetic and mechanical stresses on materials.

The Effect of Magnetic Fields on the Toughness of Overtempered 9Ni Cryogenic Steel

Commercial N.K.K. 9Ni cryogenic steel was given a 3-step heat treatment to produce a thermally unstable austenite:

1000°C 3hrs / WQ ,   800°C 1.5hrs / WQ ,   600°C 250hrs / WQ

After cooling to room temperature, these specimens had about 18 vol.% austenite phase, and a room temperature Charpy toughness of 205J. We found that 8.5 % austenite transformed to martensite upon cooling to liquid nitrogen temperature, causing a 63J decrease in the room temperature impact toughness. In the present experiment the austenite content and Charpy data were compared for specimens which had: 1) only been cooled in liquid nitrogen, and 2) which had been both cooled in liquid nitrogen and subjected to intense magnetic fields. The high magnetic fields were generated by a pulsed magnet which discharges a bank of capacitors into a cold copper solenoid. Prepared Charpy impact specimens were immersed in the liquid nitrogen in the bore of the solenoid, and were subjected to a series of seven magnetic pulses with peak fields increasing from 12 to 16T. The pulses had a rise time of approximately 0.01 sec followed by a slow exponential decay. Soviet work [6,7] with the application of even more rapid and intense magnetic fields to steel specimens has shown only small (a few °C) effects due to eddy current heating and inhomogeneous magnetic field penetrations.

The amount of retained austenite in all samples was determined by backscatter 14.41 keV Mössbauer spectrometry. A difference procedure [8] was used to compare the austenite contents of the specimens with and without exposure to the magnetic pulses. These measurements showed a retained austenite content of 9.5 vol.% after cooling to 77K, and revealed that an additional 1.3±0.5 % of the austenite was transformed by the magnetic field. The Charpy impact toughnesses of these specimens were then determined at both room temperature (293K) and at 77K. Four or five specimens of each type were broken at each temperature in order to determine the standard deviation of the data, which was about 3J. The mean impact toughnesses of the thermally cycled specimens were 142J at room temperature, and 75J at 77K. The mean impact toughnesses of the thermally cycled specimens which were also magnetically pulsed were 130J at room temperature, and 65J at 77K. The exposure to the magnetic pulses caused a reduction in Charpy impact energy of 10 - 12J at both temperatures. It seems reasonable to attribute this additional loss of toughness to the additional martensite transformation induced by the magnetic field: each 1 % reduction in austenite content corresponded to the same 7J decrease in impact toughness when the austenite was either thermally or thermomagnetically transformed.

No effect of pulsed magnetic fields was observed for the Charpy impact toughness of N.K.K. 9Ni steel with the QT heat treatment, nor in Nippon 6Ni QLT steel. There was no thermal or thermomagnetic transformation of the austenite in these materials, either.

The Effects of Magnetic Fields on 304 Stainless Steels

First, standard 6.4mm tensile specimens of 304L and 304LN stainless steels were pulled different amounts in order to determine the quantity of strain-induced martensite formed at 77K and at room temperature without the magnetic field. X-ray diffractometry results are shown in Fig. 3. Long tensile specimens with a length of 153mm and a diameter of 3.2mm were constructed for straining in the pulsed magnet described above. The straining fixture is shown in Fig. 4. The top end of the specimen was threaded into the loading bolt. The specimen was then strained at 77K by torquing a nut on the loading bolt (nut not shown) against the flat loading surface of the front support. A slotted key on the unthreaded section of the loading bolt eliminated torsional forces. The lower end of the specimen was threaded into a stationary end support and immersed within the bore of the magnet. The gauge length of the specimens was marked into three 51mm segments for elongation measurements. These segments experienced identical mechanical stresses and temperatures, but only the lower segment was exposed to intense magnetic fields. Several specimens were sacrificed for calibration of the bolt loading fixture, and measurements of

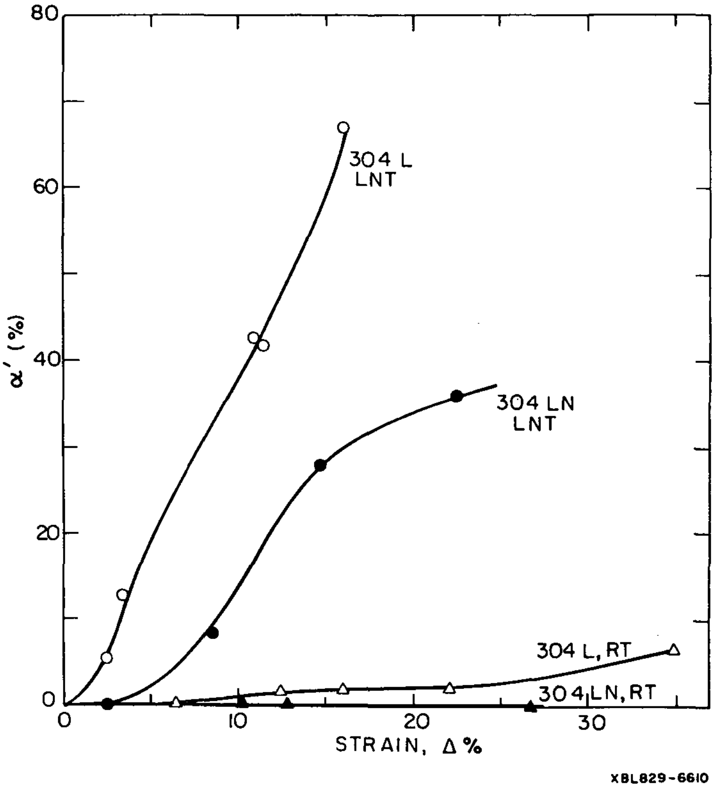
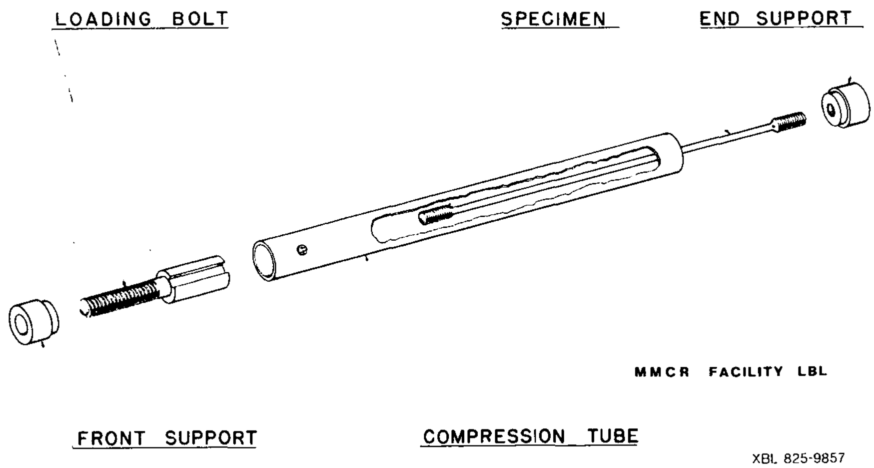


Fig.3 Strain-Induced Martensite in 304L and 304LN

Fig.4 Straining Fixture for Pulse Magnet

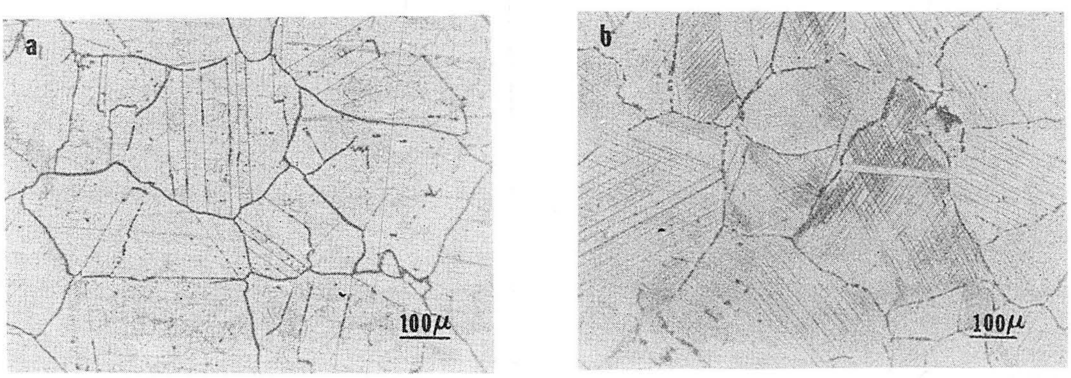


torque versus turns of the loading nut showed that the tensile force was reproducible within 5 %.

When specimens loaded about halfway into the elastic range were subjected to 16T magnetic pulses, no observable martensite transformation nor plastic deformation was found. However, clear effects of the 16T magnetic pulses were observed with specimens that were loaded into the plastic range at 77K. A time-dependent load (torque) relaxation was observed for both 304L and 304LN specimens which were deformed plastically. This creep behavior has been noted previously [9,10] in experiments without magnetic fields. However, when the specimens were exposed to a set of seven 16T magnetic pulses, the load relaxation was observably greater after the same 5 min time interval. The specimen elongation was not uniform, the 51mm segment in the bore of the magnet always showed more elongation than the other two segments. For 304L specimens which had been gradually pulled to an overall elongation of 1.5 to 2 % while exposed to 16T pulses at 77K, the ratio of the fractional elongation of the lower segment to the fractional elongation of the upper segment was 1.9. In 304LN specimens pulled a smaller amount, the lower end of the specimen showed an elongation 1.3 times that of the upper end.

Subsequent x-ray and optical metallographic analysis showed that both the volume fraction of martensite and the mode of plastic deformation was affected by the magnetic field. The 304L specimens contained about 6 vol.% martensite in the segment in the magnet versus 3 % martensite in the upper segment. The martensite volume fraction has not yet been quantified in the 304LN specimens, but the partial martensite transformation is evidenced by a magnetic permeability >0.01 in the lower segment, and a magnetic permeabil-

Fig.5 Optical Micrographs of 304L Specimen Strained a Total of 2 %
left: Segment out of Magnet right: Segment in Magnet



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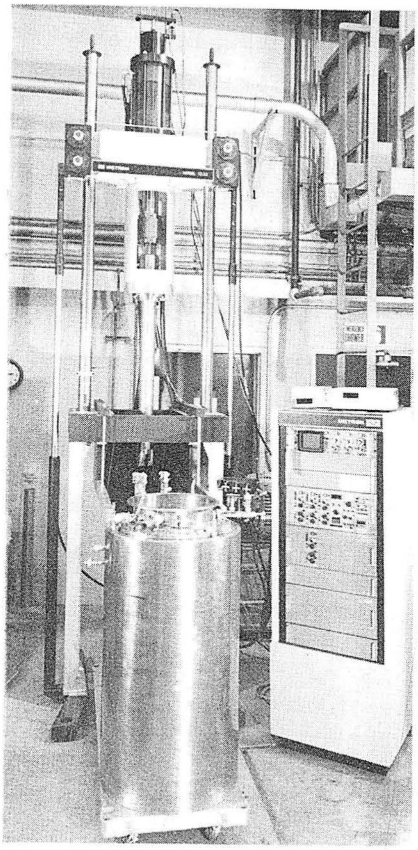
ity <0.01 in the upper segment. 304LN steel, with its more stable austenite (see Fig. 3), shows smaller magneto-mechanical effects than 304L steel. Optical metallography showed that deformation in 304L stainless steel exposed to the magnetic field involved the formation of a dense distribution of a large number of well-defined slip lines, while deformation in the material outside the intense magnetic field was much more uniform with fewer slip lines (see Fig. 5).

In both alloys the excess elongation of the lower segment of the specimen is too large to be entirely explained by the volume dilatation associated with the martensite transformation. This suggests that the martensite transformation may promote slip in the surrounding matrix to enhance its mechanical effect. These results demonstrate that the presence of high magnetic fields in addition to plastic strains enhances the martensite transformation and modifies the deformation mode in 304L and 304LN stainless steels.

Design and Construction of the LBL Magneto-Mechanical Cryogenic Research (MMCR) Facility

Research into the mechanical properties of materials in an environment resembling that of a superconducting magnet requires a versatile facility capable of cryogenic tensile, toughness, and fatigue crack propagation measurements in the presence of large, steady magnetic fields. Well-characterized fatigue and toughness tests require large specimens and therefore a large bore magnet. The superconducting magnet system for the MMCR facility was fabricated by the Magnetic Corporation of America, and can be seen in Figs. 6 and 8. Its NbTi solenoid has been successfully operated at 8.6T, and it is still training. The variable temperature anti-cryostat that encloses the specimen includes a large 105mm diameter tail section within the solenoid. The cryostat/magnet rolls on tracks under the lower crosshead of a modified 244kN Instron model 1332 servohydraulic load frame. The hydraulic actuator assembly is mounted on the upper crosshead, and in our design there are no forces acting between the upper crosshead and the support frame. Consequently we were able to employ the hydraulic lift cylinders to immerse or retract the specimen and mechanical linkage from the cryostat. In addition, it is possible to maintain a steady, temperature-independent pre-load on the specimen during these operations, thereby eliminating uncontrolled stresses on the specimen due to different rates of thermal expansion in the mechanical linkage.

One practical measure of success of any 4K mechanical properties test facility is the amount of liquid helium consumed during testing. Both the cooldown and the steady-state liquid helium consumptions were minimized by using a long and narrow cryostat to



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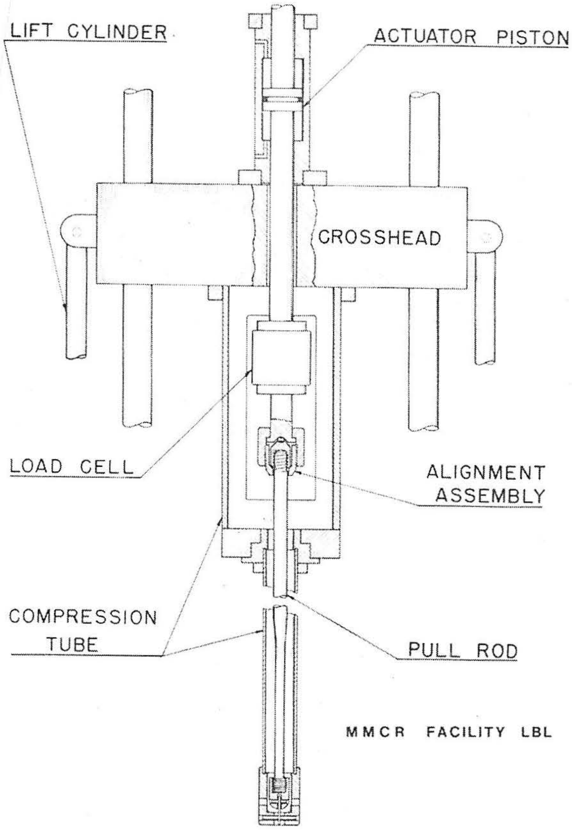

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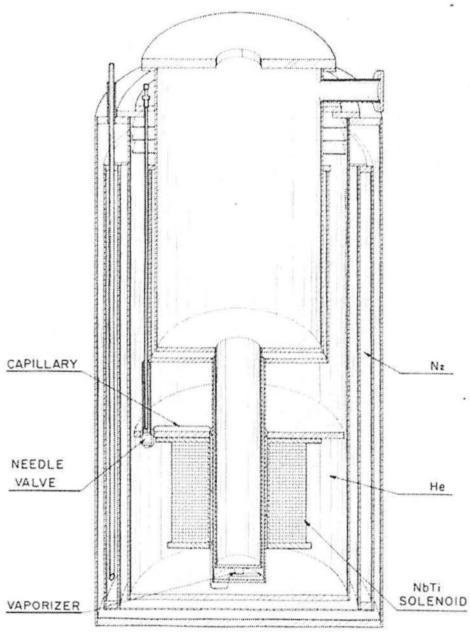

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Fig.6 Photograph of MMCR Facility

Fig.7 Mechanical Linkage

Fig.8 Superconducting Magnet

efficiently utilize the enthalpy of the expanding helium gas. Heat conductivity down the mechanical linkage is also reduced by its length and narrow cross sectional area. Since the yield strength of the 310 stainless steel cryogenic pull rod is strongly temperature-dependent below room temperature, the lower end of the pull rod has only half the cross sectional area of the room temperature end. The compression tube was made as wide as possible (89mm diameter) so that stability against buckling could be achieved with the narrowest wall thickness. After a 77K pre-cooling of the specimen and mechanical linkage, the cooldown to 4K in our general purpose Janis liquid helium cryostat requires about 12 liters of liquid helium. The steady-state liquid helium consumption with this system is sufficiently low to make long fatigue tests practical: approximately 12 liters/hour.

The long mechanical linkage shown in Fig. 7 requires an alignment assembly to ensure that no bending moment is applied to the specimen. The alignment assembly works as a ball joint where two angular degrees of freedom are provided by the free motion of a convex spherical surface connected to the pull rod, and a mating concave spherical surface connected to the load cell. This alignment assembly also allows small compressions of the pull rod, a feature which adds versatility and also provides some protection for the system.

Although it can bear 244kN loads, our long and narrow mechanical linkage has a large mechanical compliance. In fatigue testing the linkage behaves as a damped mechanical oscillator. Strain-controlled fatigue testing will largely compensate for this compliance, but such behavior causes a phase difference between the applied stress and the specimen strain. This effect is no more than 1^{o} at 20Hz.

Conclusion

The mechanical behavior of structural steels in the presence of high magnetic fields is an issue of importance to high field superconducting magnet development, and may become even more important as increasing performance demands that these materials sustain larger stresses. New experimental data show some effects of high magnetic fields on the mechanical properties of steels containing metastable austenite. Although there are now many questions as to what engineering implications these effects may have, answers should be available soon.

Acknowledgments

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