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MAGNETIC SUSCEPTIBILITY AND STRAIN-INDUCED MARTENSITE FORMATION AT 4 K IN TYPE 304 STAINLESS STEEL

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ABSTRACT

Changes in magnetic susceptibility, χ, as a function of strain-induced structural transformations in AISI type 304 stainless steel at 4 K have been observed using a mutual inductance technique with simultaneous measurement of stress and strain. There is a small increase in χ coincident with plastic strain and a large increase in χ with the load drops that occur during serrated yielding. These are attributed to the formation of bcc martensite. The increases in χ are irreversible upon unloading. The application of a moderate 3-MA/m (37-kOe) dc field had no effect on the martensite formation.

INTRODUCTION

Plastic deformation of some metastable austenitic stainless steels, such as AISI type 304, at cryogenic temperatures causes localized transformations to martensite. This phase is ferromagnetic, with a large coercivity at 4 K. These alloys are also known to exhibit serrated yielding at liquid helium temperatures.[1] The first measurement of magnetic susceptibility during plastic deformation was by Kelha and Niinikoski.[2] Bolshutkin et al. investigated susceptibility correlated with martensite transformations at cryogenic temperatures.[3] In the present work, quantitative measurements of susceptibility changes at 4 K during martensite formation, with and without a background field, are reported. The results show that most of the martensite formation occurs during the serrated yielding, and that the amount formed increases with the magnitude of the load drops.

EXPERIMENT

Tensile specimens with about 6-mm gauge diameter and 4-cm reduced-section length (cf: Ref 4) were machined from type 304 stainless steel rod stock. Composition (by weight) was nominally 73% Fe, 18% Cr, and 9% Ni. The specimens were annealed in helium gas at 1066°C for 15 min, quenched in water, and electropolished to remove 0.1 mm from the diameter. Tension was applied with the specimen immersed in liquid helium within the bore of a 3-MA/m (37-kOe) superconducting magnet. Magnetic ac susceptibility, χ, could be measured with the dc field off. When the dc field was on, it saturated the specimens and precluded measurement of χ.

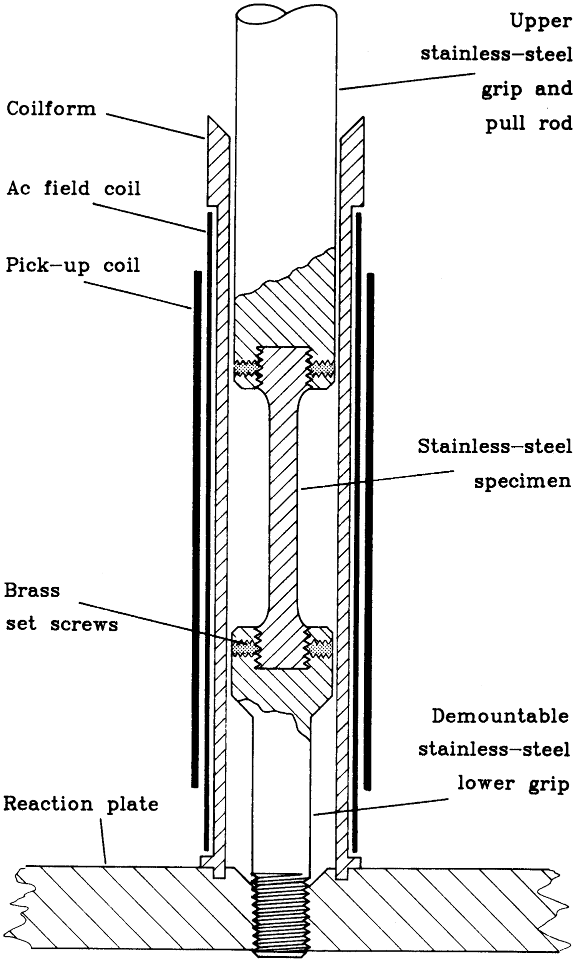

Fig. 1. Construction of susceptometer and demountable grips. Reaction tube not shown.

The unfractured specimens could be unscrewed from the reaction plate and, while still attached to the pull rod, withdrawn from the Dewar. This allowed the samples to be changed without warming the magnet and reaction-tube assembly. The construction of the susceptometer is shown in Fig. 1. It was calibrated with a standard of dimensions similar to those of the specimens. The calibration was verified by numerical methods. Note that the entire specimen is well within the pick-up coil and remains so even after straining.

A mutual inductance technique was used to measure χ. A schematic of the electronics is shown in Fig. 2. An ac oscillator, in constant current mode, drives the field coil and provides a reference for the lock-in amplifier, the phase-lock phase-shifter, and the oscilloscope. When a magnetic transformation in the specimen occurs, the mutual inductance between the field coil and the pick-up coil increases. The constant ac current, I, ensures a constant field even when the self-inductance of the field coil increases. The emf induced in the pick-up coil is input to a differential amplifier. The high input impedance of the amplifier

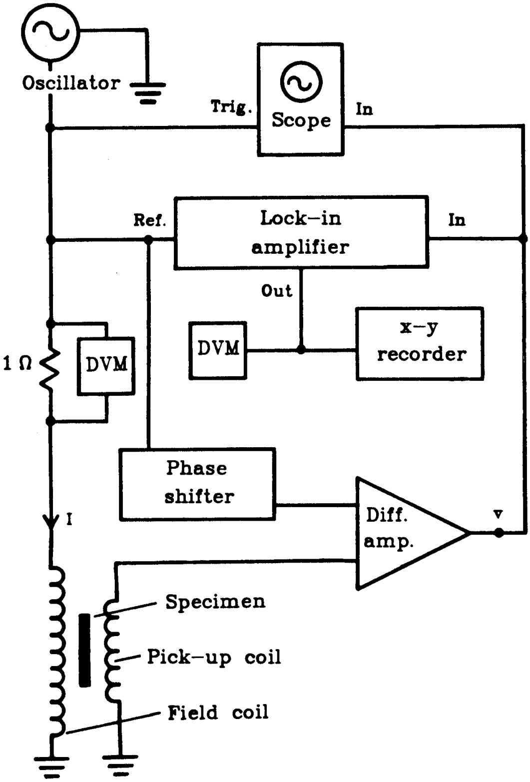

Fig. 2. Electronics schematic for susceptibility measurements.

ensures that substantially no current flows in the pick-up coil so that the emf is about equal to its open-circuit value. Also connected to the differential amplifier is a phase shifter. This specially designed circuit locks onto the frequency of the driving signal. Its output, variable in amplitude and phase, is set to null the output of the differential amplifier, v, after the specimen is inserted but before it is strained. The usual method of compensation with an identical coaxial compensating (or "bucking") coil was not chosen because of the limited space in the Dewar. The output of the differential amplifier is connected to a lock-in amplifier. The dc output of the lock-in was recorded vs. stroke (strain). Load (stress) was also recorded vs. stroke.

The frequency selected for the ac χ measurement was 100 Hz. For the specimen geometry, permeability, and resistivity, this frequency was not high enough to cause eddy-current problems.[5] The field coil was operated at 4 kA/m (50 Oe) rms. This relatively large ac field was needed because of the large coercivity of martensite at 4 K.

RESULTS AND DISCUSSION

Figure 3 is a plot of χ and load (stress) vs. stroke (strain). Stroke was controlled at 0.1 mm/min. The specimen was unloaded at various points on the curve to see whether the increased magnetic signal was reversible. In the initial elastic and plastic regions (a, b, and c in Fig. 3), there was a small, reversible increase in χ (d). In the plastic region prior to each serrated yield (e), there was a small irreversible increase in χ (f). Coincident with each of the serrated yields (i), there was a large, irreversible increase in χ (j) caused by the formation of strain-induced bcc martensite, which is ferromagnetic. Immediately following the load drops, as reloading occurred (o), the increase in χ was once again reversible (p). The slope of this increase became larger as the number of load drops increased. The reversible increases in χ may be attributed to the inverse positive magnetostrictive effect: an increase in χ due to stress,[6] predominantly of the already transformed martensite.

The martensite formation at low temperature in metastable austenitic stainless steels is strain induced.[7] The magnitude of the increase in χ, and therefore the amount of martensite formed, correlates with the magnitude of the strain (load drops). This relationship is plotted in Fig. 4 for two specimens strained at the usual rate of 0.1 mm/min. A third specimen was strained at the rates of 0.2 mm/min and 1.2 mm/min. The increase in χ depended on the magnitude of the load drop but not on the strain rate. Because of the adiabatic conditions at 4 K during the sudden load drops, large localized rises in temperature occurred. It is likely

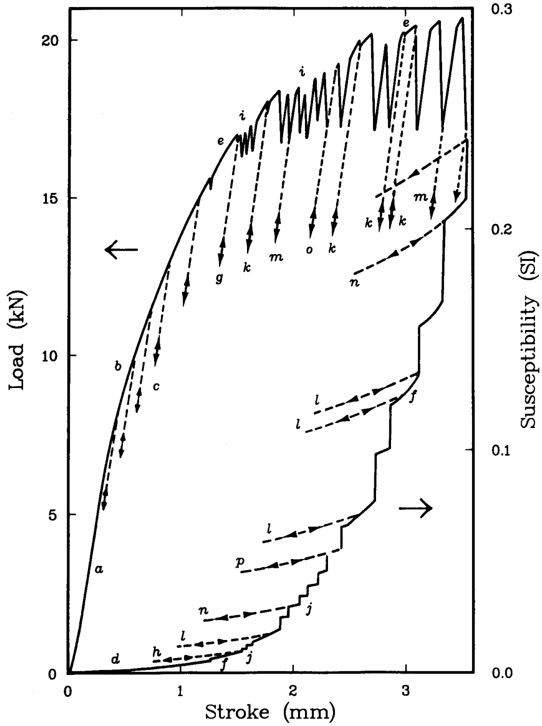

Fig. 3. Load and increase in volume susceptibility vs. stroke: (a) initial elastic region, (b) initial plastic region, (c) unloading to zero and reloading, (d) reversible increase in χ during a, b & c, (e) plastic region prior to load drop, (f) irreversible increase in χ during e, (g) unloading and reloading prior to second load drop, (h) reversible decrease and increase in χ during g at higher level than d, (i) serrated yielding, (j) irreversible increases in χ during i, (k) unloading and reloading prior to load drop, (l) reversible decrease and increase in χ during k, (m) unloading and reloading immediately following load drop, (n) reversible decrease and increase in χ during m, (o) elastic loading, unloading, and reloading following load drop, (p) reversible increase, decrease, and then increase in χ during o.

that the nonlinear relation between the magnitudes of the load drop and the increase in χ reflects the isothermal nature of the transformation of martensite in austenitic steels: larger temperature increases are associated with larger load drops, and larger amounts of martensite form at higher cryogenic temperatures.[1]

A 3-MA/m (37-kOe) dc field had no effect on the amount of martensite formed. A comparison was made of four specimens, two strained with the background dc field off and two with it on. The ratio of χ after straining divided by percent strain (about 6%) was virtually the same for all four specimens. Another specimen was strained until it was in the serrated yielding region. After each load drop, the specimen was unloaded, the field alternately applied or removed, and the specimen reloaded until the next load drop. The increase in χ was recorded for 10 cycles and did not depend on the presence or absence of the field.

Although it does not apply to the above results, one possible source of error in measuring the effects of magnetic fields on mechanical properties is that the application and removal of a dc field may exert a force on the reaction tube or on the pull rod, which results in changes in load. This could lead to spurious conclusions regarding transient magnetic field effects in the

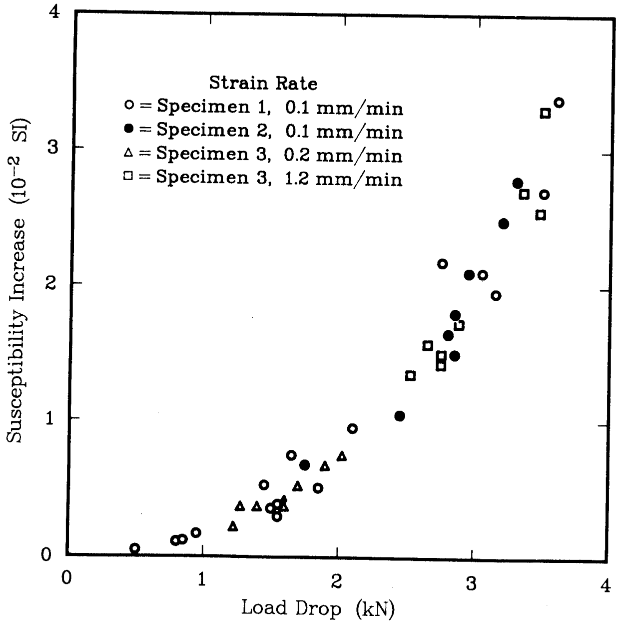

Fig. 4. Increase in volume susceptibility as a function of magnitude of load drop.

specimens. Figure 5 shows load and magnetic field vs. stroke (controlled as a function of time) in the elastic region of a specimen (in this case austenitic type 310 stainless steel, nominal composition: 54% Fe, 25% Cr, 21% Ni). No changes in the stress-strain curve due to transient field conditions nor significant magnetostriction would be expected in the elastic region of this low-magnetization, stable alloy. However, as the field increased, the load increased above its trendline. A reduction in field caused the load to return to its trendline. The magnet was quenched after its final charge, causing a precipitous drop in load. These apparently transient effects in the specimen were actually due to the interaction of the magnetic field with the weakly magnetic reaction tube and pull rod and local magnetic fields caused by eddy currents. Such effects should be considered in transient-field experiments (cf: Ref. 8).

CONCLUSIONS

AISI type 304 stainless steel, when plastically strained at 4 K, is known to undergo serrated yielding. This results in a pronounced increase in χ, as measured at room temperature, which has been attributed to the transformation of fcc austenite (γ phase)

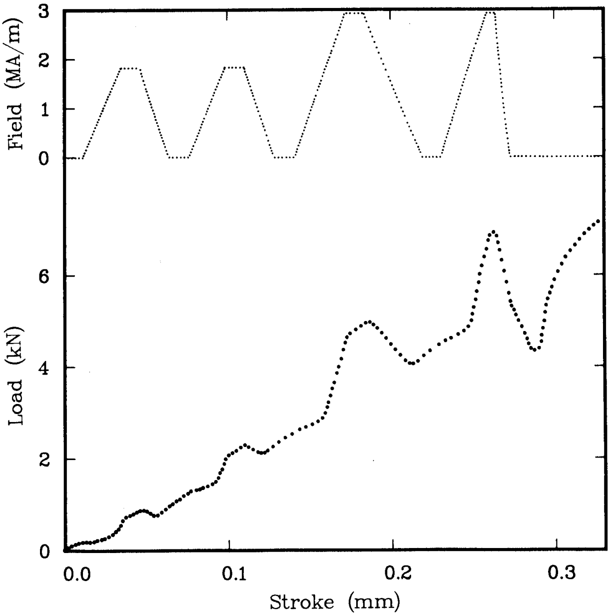

Fig. 5. Artifacts in load-stroke curve caused by application and removal of magnetic field.

to ferromagnetic bcc martensite (α' phase). In this work, the ac χ was measured at 4 K using a mutual inductance technique while simultaneously measuring stress and strain. The following results were obtained.

1. There was a small increase in χ in the initial elastic and plastic regions, which was reversible upon unloading.
2. There was a small, irreversible increase in χ with plastic deformation prior to serrated yielding. This is attributed to the formation of strain-induced martensite.
3. There were large, irreversible increases in χ coincident with the load drops associated with serrated yielding. These are also attributed to the irreversible formation of strain-induced martensite. The magnitude of the increases in susceptibility correlated with the magnitude of the load drops.
4. Immediately following a load drop in the serrated yielding region, there was a reversible increase in χ as elastic reloading occurred. The slope of this increase became larger as the number of load drops increased. This may be attributed to the inverse magnetostrictive effect. After reloading, further plastic strain prior to the next load drop caused a small, irreversible increase in χ.
5. The application of a moderate, 3-MA/m (37-kOe) dc field at 4 K had no effect on the formation of martensite.
6. The application and removal of a dc magnetic field caused changes in the stress-strain curve when measured as load vs. stroke. These changes were likely due to the magnetic force on the reaction tube and pull rod rather than transient magnetic effects in the specimen.

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