INVESTIGATION OF DEFORMATION USING SQUID MAGNETOMETRY

R. B. Mignogna and H. H. Chaskelis

Mechanics of Materials Branch
Naval Research Laboratory
Washington, DC 20375-5000

INTRODUCTION

Most of the effort in the area of ferromagnetism has been directed towards improving magnetic properties of materials. For example, residual stress (strain) and defects are studied for their influence on magnetization, in order to produce magnetically hard materials etc., as opposed to studying the influence of magnetic properties of materials on their mechanical behavior or as a means of investigating micro-mechanical behavior. A few exceptions are studies of macro-mechanical behavior under very high magnetic fields [1], the "delta E" [2] and Barkhausen effect [3-5]. These are directed towards understanding mechanical behavior but usually use intrusive means, by perturbing the material with a magnetic field to produce measurable changes. A great deal of interest has been shown in the Barkhausen effect by the NDE community. Although this is a nondestructive technique, it is still somewhat intrusive.

The ability to measure stress/deformation, in situ, has been sought for many years. New approaches are needed to yield additional information for the nondestructive measurement of stress and understanding of deformation processes as they occur on the local scale. Weinstock and Nisenoff [6], using a SQUID (Superconducting QUANTUM Interference Device) gradiometer, reported changes of the magnetic field near a steel specimen during tensile deformation, i.e. elastic and plastic deformation. No external field was applied during the test (except the earth's magnetic field). Although the report was very cursory, it indicates the potential of using SQUID magnetometric techniques to observe weak magnetic fields generated by magnetoelastic effects as a passive and non-intrusive method of studying deformation. The recent advances in superconductivity and resulting technological advances of the SQUID devices imply even greater future potential in terms of sensitivity, spatial resolution and utility (liquid nitrogen versus liquid helium devices).

The purpose of this work is to re-examine and extend the results presented in [6] to gain better insight into the real potential of using SQUID magnetometry for NDE with applications for stress or deformation measurements. Preliminary results of this study are presented here, comparing flux gate magnetometer and SQUID gradiometer measurements made during tensile deformation of cold rolled steel and 99% pure nickel.
MAGNETOElastic interactions are due to coupling of strains and the magnetic properties of the material, usually broken down into the following topics: exchange energy, magnetoelastic energy, anisotropy energy and magnetostatic energy. The tendency of ferromagnetic materials to seek the lowest energy state results in an interaction between the various energies and the formation of domains. The domains are regions in the material in which all of the magnetic moments are aligned in the same direction. The direction and boundaries of the domains arrange themselves in such a manner as to make the total energy a minimum.

The exchange energy arises from electron spin motion. The anisotropy or magnetocrystalline energy can be described as directional effects of spin-orbit coupling reflecting the symmetry of the crystal by interaction of the lattice atoms. This results in the tendency of magnetization to lie along certain definite crystallographic directions, called directions of easy magnetization. Magnetostatic or 'self-energy' is due to the interaction of the material with its own magnetization as opposed to an external field. The magnetoelastic energy is due to the interaction between the magnetization and mechanical strain of the lattice, termed magnetostriction.

Magnetostriction is manifest in the observations (first by Joule 1842) [7] of changes in length or strain due to changes in magnetization. An inverse effect is also observed (the Villari effect) [8], application of a stress will change the magnetization. Magnetostriction results because of the dependence of the anisotropy energy on strain. This dependence is such that the stable state of the material is deformed with respect to the base symmetry of the lattice. Spontaneous deformation occurs so as to lower the anisotropy energy.

Another effect attributable to magnetostriction is the so called 'delta E' effect. This is an apparent change in Young's modulus as a function of magnetization. Changes of modulus of almost 20% have been reported for nickel [8] and 1000% (thousand) for amorphous thin films [2]. This effect is due to the change in 'total' domain alignment and the accompanying strain associated with the anisotropy energy.

As demonstrated by hysteresis during magnetization by an applied field, these processes can be highly irreversible. Changes in the stress or applied field can result in reversible domain growth or rotation or it can result in an irreversible transition of the domains. Extremely rapid "jerky" transitions are believed to be the cause of the Barkhausen effect (noise, measurable by both magnetic and acoustic means).

EXPERIMENTAL ARRANGEMENT

A schematic diagram of the experimental system is presented in Fig. 1. The specimens ("dogbone" geometry) were subject to tensile deformation in a vertical (load axis) testing machine. The use of a vertical testing machine constrained the positioning of the SQUID gradiometer (referenced only as the SQUID, henceforth). The SQUID was in an "open" helium dewar and the gradiometer axis was along the dewar axis. The gradiometer coils are located in the "tail" (small end) of the dewar and are approximately 6.3 cm long. Because of the constraints of the vertical machine and open dewar, the SQUID was aligned at 45° to the tensile axis with the gradiometer coils approximately 25 cm from the specimen, as shown in Fig. 1. At this position, it was still possible to operate the SQUID on its least sensitive setting. The flux gate magnetometer (referred to as the magnetometer) was positioned on the opposite side of the specimen with its
axis at 45° in order to "mirror" the SQUID. However, because of poor signal to noise, it was necessary to place the magnetometer approximately 5 cm from the specimen. The probe of the magnetometer was approximately 7.0 cm long. The distances given are measured from the center of both the magnetometer probe and SQUID coil to the specimen.

The gage length of the steel specimens was 17 cm long, 1.7 cm wide and 0.15 cm thick. Steel specimens were machined from sheet material, along both the rolling and transverse direction. Nickel specimens were machined from strip material along the rolling direction. The gage length of the nickel specimens was 13 cm long, 0.6 cm wide and 0.3 cm thick. Tensile load was applied at a fixed rate of 0.05 cm/min (crosshead speed).

EXPERIMENTAL RESULTS

For the most part, the data observed from both the SQUID and magnetometer were in very good qualitative agreement with each other. The types of changes displayed by the SQUID were also observed with magnetometer. This can be seen in Figs. 2 through 5, not considering overall positive or negative directions (Fig. 5). Because of this similarity in response only the SQUID data will be described.

Figure 2a shows the engineering stress versus change of time for a steel specimen. The data shows the specimen being initially loaded elastically, followed by yield, some plastic deformation followed by immediate unloading (all together referred to as a loading cycle). A short time later the specimen was reloaded to approximately one half of the yield stress and then unloaded. Figures 2b and 2c present the data obtained from the magnetometer and SQUID, respectively, also as a function of time. The time axis is the same for all three sets of data shown in Fig. 2-5.
During the first loading cycle, as the elastic load increased, a decrease in magnetic field was observed by the SQUID. At approximately 60% of yield the field began to increase again or "reverse" (this change from decreasing to increasing magnetic field or vice versa will be referred to as "reversal"). Upon reaching yield the field plateaus to some extent, as does the stress. During unloading, the changes observed in the field mirror, for the most part, the changes observed during loading. These results are in good qualitative agreement with those of Weinstock [6].

The second loading cycle was performed by observing the SQUID output. When the change in magnetic field began to level off, implying the "reversal", the crosshead was stopped and then reversed. As can be seen in the second loading cycle of Fig. 2c, the "reversal" begins to occur at about 60% of yield.

Results obtained during tensile deformation of a nickel specimen are shown in Fig. 3, using the same display format as Fig. 2. During the first loading cycle, the specimen was continuously deformed beyond yield, the crosshead was then stopped and held for a few seconds and then unloaded. Some relaxation can be observed in Fig. 3a (first loading cycle). The
Fig. 3. Nickel specimen. (a) Stress versus time. Changes in magnetic field versus time (b) magnetometer and (c) SQUID. Second loading cycle was similar to the first. However, once the crosshead was stopped, the load was held for a longer period of time, followed by some additional loading and then unloading. Relaxation can be seen in both the stress and SQUID measurements during the "hold" period of the second loading cycle. It should be noted that the "reversal" observed for steel during elastic loading did not occur for the 99% pure nickel specimens.

Another steel specimen was loaded in "steps". The deformation rate was the same as stated earlier but in this case the crosshead was halted at various stress levels (elastic), held for a few seconds then the load was either increased or decreased. Some perturbative cycling about a fixed load was also performed in the elastic regime. The specimen was then loaded beyond yield for a short time, partially unloaded and immediately reloaded, again beyond yield. The stress versus time data is shown in Fig. 4a. The changes in magnetic field follow the changes in stress rather well below about 60% of yield. Comparison of stress and changes in magnetic field become more complicated beyond approximately 60% of yield due to the "reversal".

There appeared to be a type of hysteresis occurring, as can be seen after the first elastic unloading step of Fig 4. The stress level at this step is only slightly above the stress level at the first load step, but
Fig. 4. Steel specimen, step load. (a) Stress versus time. Changes in magnetic field versus time (b) magnetometer and (c) SQUID

the change of magnetic field lags and is still below the change produced by the second load step.

The same type of "step" loading was used for a nickel specimen, shown in Fig. 5. These results are quite similar to those just described for steel. The hysteresis, described in Fig. 4, was also observed in the nickel but the complications due to the "reversal" in steel are not present. Although the field changes are qualitatively the same as the stress changes, the magnetic field changes become progressively less or "compress" for a given change of stress as the yield stress of the material is approached. The "compression" can be easily seen by comparing Fig 5a. to either Fig. 5b or 5c as the yield stress is approached. The "compression" was also observed for the steel specimens but was less obvious due to the "reversal" that occurs. The reason Fig. 5c is the negative of Fig. 5b is that magnetometer was rotated 90° counter clockwise with respect to the position shown in Fig. 1 for this particular specimen.
Fig. 5. Nickel specimen, step load. (a) Stress versus time. Changes in magnetic field versus time (b) magnetometer and (c) SQUID

SUMMARY OF RESULTS

As mentioned previously, these are preliminary results and although definite conclusions cannot be made, a number of interesting features have been observed. The changes in magnetic field as a function of tensile deformation of steel qualitatively agrees quite well with the results of [6]. The magnitude of the magnetic field change for cold rolled steel deformed along the rolling direction was approximately twice the change observed during deformation along the transverse direction.

The "reversal" observed in steel was not observed for the 99% pure nickel specimens. Below approximately 60% of yield, the change of magnetic field "tracked" the change of stress for both materials. Above 60% of yield, changes of magnetic field per unit change of stress decrease rapidly as yield stress is approached.

Hysteresis was observed when the loading direction was reversed (increasing load versus decreasing load). In addition some initial history dependence was noted. This history dependence appeared to decrease with both total strain.
DISCUSSION

Magnetoelastic interactions are complex and interwoven, as briefly discussed earlier. A number of issues must be considered: material, texture, residual stress (macro and micro), specimen geometry, initial magnetic state, history dependence including reversible and irreversible processes, load configuration i.e. tension, compression, torsion, etc., and the fact that the magnetic field is a vector. Most of these issues are rather obvious, the materials iron and nickel, for example, have different crystal anisotropy and magnetostriction constants (for both types of constants, iron is positive and nickel is negative, except for k2) [9]. This results in different "easy" magnetization directions for the two materials, [100] for iron and [111] for nickel. Because of these intrinsic material differences, tensile deformation of iron should preferentially align domains parallel with the tensile axis, whereas nickel domains should align perpendicular. A number of these "points" were considered, to some extent, during these measurements. Those that were not accounted for are: specimen geometry, initial magnetization and residual stress.

The fact that the magnetic field is a vector indicates the possibility of ambiguity in the measurements presented here. Both the flux gate magnetometer and SQUID gradiometer used for this work measured only one component of the magnetic field. This implies that the changes observed in magnetic field may have been caused by a change in direction, change in magnitude or a combination of both direction and magnitude. To remedy this situation a three, orthogonal-axes SQUID will be used for further measurements. One of the most intriguing questions is in regard to the "compression" of the magnetic field changes near the yield stress. Whether these changes are due to field rotation or magnitude change or both, there appears to be either some relation to yield stress or just an unfortuitous effect at these stress levels.

REFERENCES