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Detection of plastic deformation in structural steel using scanning SQUID microscopy

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Abstract
The magneto-mechanical behaviour of structural steel specimens stressed up to a plastic deformation stage using a high-T_c scanning SQUID (superconducting quantum interference device) microscope is investigated. The correlation between the gradient of the normal component of the magnetization and dislocation density, before the crack initiation, is carried out. The capability of scanning SQUID microscopy to detect the residual magnetization, due to the tensile stress, with a non-invasive technique is reported.

Keywords: magneto-mechanical effects, microscopy instrumentation, nondestructive testing of materials, SQUID devices

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

1. Introduction

One of the most important problems in nondestructive testing is the development of techniques to detect the fatigue effect in metallic alloys during the cycle life of the material, before crack initiation.

High cycle fatigue is ordinarily divisible into three distinct stages. The first stage lasts for about the first 10% of the fatigue life, and is characterized by significant changes in the dislocation density and structure, as the microstructure accommodates to the cyclic stress or strain. The second stage extends through roughly the next 80% of the fatigue life. In this stage the dislocation substructure evolves slowly and becomes increasingly heterogeneous, leading to the formation of the persistent slip band that will eventually nucleate cracks. The third stage begins with the formation of the fatigue cracks at about 90% of the fatigue life and ends with final failure.

Detectable crack nucleation does not occur, generally, before the material has experienced 90% of the fatigue life. Since fatigue cracks can propagate under loads that are small compared to those required to nucleate them, and since crack growth accelerates geometrically as it goes further, the time interval for crack detection between nucleation and catastrophic failure may be very short.

Because significant microstructural changes occur in the first and the third stages of fatigue, researchers have had some success in developing NDE methods probing them. Magnetic methods are particularly promising for ferromagnetic materials such as structural steels. Parameters such as the saturation magnetization, coercivity, Barkhausen noise and magnetic hysteresis change significantly during the initial and final stages of fatigue. Magneto-mechanical effect has been detected using a SQUID magnetometer, measuring changes in the magnetic hysteresis of metallic alloys and structural steel [1]. Indeed, a magnetic field sensor with high field sensitivity and spatial resolution as the scanning SQUID microscope (SSM) is requested to measure the low magnetic field signals characterizing dislocation motion. These properties are successfully satisfied by the SQUID microscope that has already been used in the detection of fatigue damage [2–7].

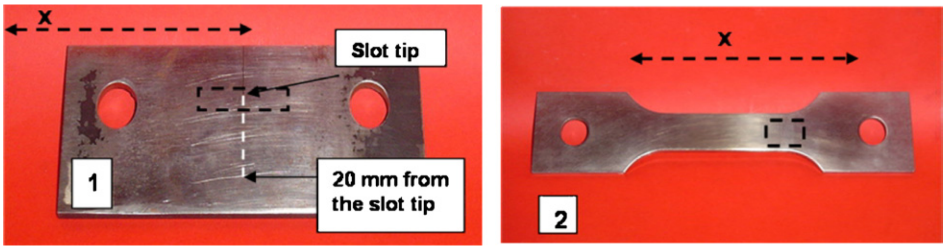

Figure 1. Test samples, the dotted area indicates the inspected region.

An interesting application of SQUID microscopy is the possible detection of early plastic deformation before the crack initiation.

The mechanism of plastic deformation in metals is due to the motion of dislocations through the crystal lattice. Dislocations interact, multiply and harden the material in the process known as ‘work hardening’. Dislocations, grain boundaries and grain orientations interact with magnetic domain walls and, therefore, may influence the magnetic structure and properties of ferromagnetic materials. The primary relevant structural alteration that occurs during plastic deformation is the increase of dislocation density. When a material is deformed, at first the dislocation density increases rapidly along with the hardness. As deformation continues, the dislocation density tends to saturate, and the hardness approaches a maximum that is retained even during further deformations. As evidenced by hardness alteration, in this process an important parameter is the incremental damage rather than the amount of deformation that the samples have experienced. The SQUID response detects a magnetic field variation only if the magnetic properties have been changed enough to alter the density of micro-structural defects that pin the domain walls.

Moreover, when the SQUID microscope is used to detect the fatigue damage in ferromagnetic materials, it can show two drawbacks. First, in the second stage of fatigue, which is the longest and usually the most important stage, the SQUID microscope does not readily measure the growth of deformation. In the second stage, in fact, the overall dislocation density is approximately constant and microstructural changes are subtle. Second, this technique does not provide reliable absolute measurement of fatigue damage. SQUID detects fatigue by measuring changes in properties rather than the properties themselves and hence, it is only useful in situations where the affected parts can be tested periodically over time.

To detect plastic deformation by SQUID microscopy, there must be associated changes in the dislocation density that are large enough to produce measurable changes in magnetic properties. Deforming materials that already contain high dislocation density, a low or no detectable magnetic effect is produced. Instead, the plastic deformation of the sample produces a rapid increase in the dislocation density. This causes significant changes in the magnetic properties that can be easily detectable by the SQUID signal.

In this work a SQUID microscope is used to monitor magneto-mechanical behaviour in ferromagnetic specimens under mechanical load. The aim of this work is to show the correlation between the magnetization and the mechanical behaviour of the structural steel samples and how the imaging of the magnetic field gradient can be useful to probe the sample plastic deformation before the crack initiation.

2. Test samples and setup

The samples used in this work were 5 mm thick structural steel (Fe 360). The sample labelled as 1 has a rectangular shape with a 0.1 mm width slot (figure 1(left)), while specimen 2 has a dog-bone shaped geometry (figure 1(right)). The two samples’ geometry is justified by the different effect induced by the applied mechanical stress on the specimens. In particular, sample 1 is used to study the initiation and propagation of a crack raised on the slot tip, while sample 2 gives the possibility of investigating the effect of plastic deformation detecting the surface slip bands.

Both samples were demagnetized and deformed plastically with a stress cycle ranging from 0 to 32 kN (sample 1) and from 0 to 40 kN (sample 2). Sample 1 experienced ten tensile stress cycles, while sample 2 was stressed with only one cycle. The mono-axial tensile stress was applied along the x-direction (see figure 1) and measured by strain gauges glued on both sides of the samples. The magnetic hysteresis due to the tensile stress cycle was measured using Hall magnetic field sensors. Then, the vertical component of the magnetic field gradient, produced by the remanent magnetization and the plastic deformation, has been measured by a scanning SQUID microscope.

3. SQUID microscope description

In this work the commercial Scanning Magnetic Microscope model 770 purchased by Tristan Inc. has been used. It utilizes a liquid nitrogen cooled $YBa_2Cu_3O_{7-x}$ dc-SQUID magnetometer mounted inside the bottom cap of a fibreglass cryostat surrounded by the cryostat’s insulating vacuum. The SQUID magnetometer measures the vertical component of the magnetic field where the magnetic field transfer function is $500\ \mathrm{nT}/\Phi_0$. The magnetic field white noise spectral density is $10\ \mathrm{pT\ Hz}^{-1/2}$ (measured at 5 kHz) and the operating bandwidth ranges from dc to 10 kHz. In order to achieve the best spatial resolution, the SQUID is positioned as close as possible to the sample; a 50 μm thick sapphire window separates the sensor from room temperature, allowing us to operate at a sample-to-sensor distance of several hundred microns.

The SQUID is read out with commercial dc-SQUID electronics (iMag Controller, Tristan Inc.) in a flux locked

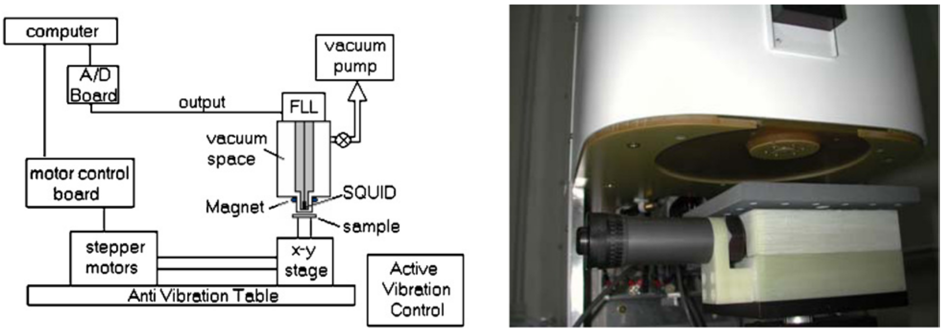

Figure 2. Scanning SQUID microscope (SSM) schema (left). The SQUID measuring head (right).

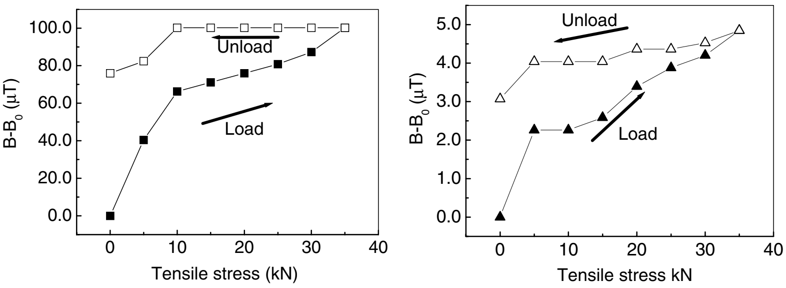

Figure 3. Magnetic field signal versus the mechanical hysteresis cycle for sample 1: (left) hysteresis loop positioning the Hall probe across the slot tip, (right) hysteresis loop at 20 mm from the slot tip. The data were normalized to the unloaded sample.

loop bias reversal mode. Two μ-metal shields enclose the overall system to eliminate environmental electromagnetic field noise, which could degrade the system performance. Moreover, to reduce low-frequency noise signals, including 50 Hz line noise, low-pass hardware and software filters are used.

Samples are positioned at room temperature on a custom non-magnetic manual control z stage (fibreglass composite) for height adjustment. The framework is positioned on a two-dimensional translation stage with a scanning range of 150 mm × 150 mm and a minimum stepper size of 12.5 μm. The translation stage is supplied with X–Y micro-tilt adjustment screws for in-plane alignment. The system is equipped with a vibration isolation air table with active dumping system. The schema of the system is reported in figure 2(left).

Samples are scanned under the SQUID sensor while a computer records the SQUID response as a function of the in-plane X–Y position, as schematically reported in figure 2(left). The scan system uses two stepper computer controlled micro-motors. Sample scanning and data acquisition are software controlled.

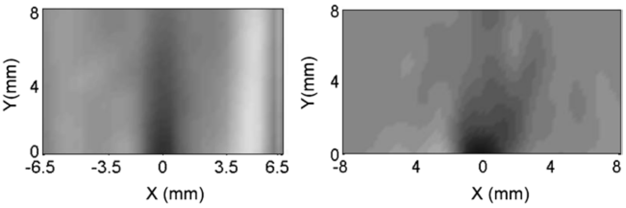

Figure 4. Imaging of steel sample 1, before the tensile stress (left) and after the mechanical load (right), as measured by the SQUID microscope.

4. Experimental results

The magnetic field response produced by the magnetization gradient due to tensile stress has been detected using a Hall probe. Two positions were chosen for the magnetic probes: at the slot tip and 20 mm from it, as indicated in figure 1(left). The results of the magnetic field measurement are shown in figure 3.

Magnetic field hysteresis is present in sample 1 after one tensile cycle up to 35 kN. Hysteresis behaviour of the magnetization with a lower intensity is also measured 20 mm far from the slot tip. It could be noted that the normal component of the magnetization, after one tensile stress cycle, is higher corresponding to the slot tip than 20 mm from it. Moreover, the area of the magnetic hysteresis loop measured at the slot tip is ten times greater than the hysteresis loop area measured 20 mm away from the slot tip.

As expected, the slot tip produces a local tensile stress concentration in the specimen. This mechanical effect is correlated to a high magnetization gradient. In other terms, around the slot tip there is a perceptible motion of dislocations, and their density decreases, moving away from the slot tip (as shown in figure 3).

To localize the plastic deformation, produced by the tensile stress, an image of the magnetic field gradient $\mathrm{d}B_z/\mathrm{d}x$ has been measured before and after the stress cycles. In figure 4 the results obtained using the SQUID microscope are shown. Before applying the tensile stress, the normal component of the magnetic field gradient appears as figure 4(left). It could be noted that there is remanent magnetization on the sample concentrated on the boundary

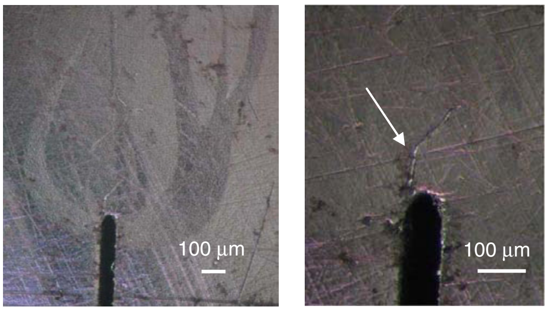

Figure 5. Optical image of sample 1 when a plastic deformation occurs (left). The white arrow indicates the crack initiation (right).

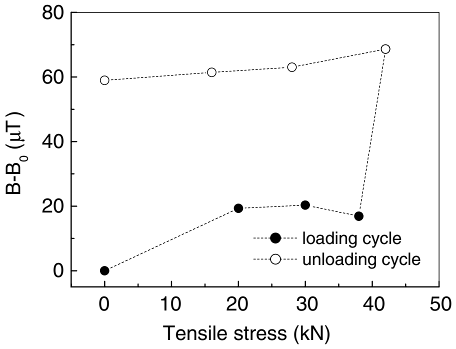

Figure 6. The magnetic hysteresis loop of sample 2, measured with a conventional Hall probe. The data were normalized to the unloaded sample.

of the slot, which is represented by the well-defined black area. This magnetization distribution hangs over the demagnetization treatment that occurs before the first tensile stress cycle. Moreover, the effect of the mechanical stress is more evident in figure 4(right), where the slot tip is surrounded by a large not well-defined dark area, which represents the plastic deformation. In our opinion the magnetic field imaging measured by the SQUID microscope allows us to detect magnetic dislocation motion arising from mechanical tensile stress.

The plastic deformation and the crack initiation across the slot tip are confirmed by an optical image of the sample, shown in figure 5. This picture shows the sample condition that is characterized by a plastic deformation and crack initiation. In figure 5(left) a circular trace, which begins from the slot tip, represents the plastic deformation stage, while the arrow in figure 5(right) indicates the crack grown initiation starting from the slot tip. This condition has been reached after ten tensile stress cycles with a maximum load of 35 kN.

Moreover, the SQUID microscope imaging can detect the effect of plastic deformation, also in the case of micro-structural changes, when no cracks are visible on the sample surface. As an example, the dotted rectangular area of sample 2, see figure 1(right), has been scanned after one tensile stress cycle, when magnetic slip bands are visible on the specimen surface.

The measurement of the magnetic field intensity, carried out for specimen 2 using a Hall probe, shows hysteresis behaviour of the structure (see figure 6), even if only one stress cycle occurs. It means that the magnetization of the sample is affected by the tensile stress applied in an irreversible way. To detect the magnetic field variation due to the magnetic dislocation motion, magnetic imaging of specimen 2 has been carried out. In particular, the scanning has been carried out on a sample area with visible slip bands.

In figure 7 the magnetic image, corresponding to some slip bands, is reported. The magnetic field gradient $\mathrm{d}B_z/\mathrm{d}x$ shows the two slip bands, which generally are called persistent slip markings (PSMs), and they appear on the structure surface by means of intrusions. Typically, the slip bands precede the initiation of the fatigue cracks after plastic deformation. The magnetic field gradient reported in figure 7 detects the presence of the slip marking by means of the dislocation density. The SQUID microscope inspection, thanks to its high sensitivity to low magnetic field and good spatial resolution, allows us to detect and localize the band positions. Since the slip bands are visible on the sample surface too, the improvement of this technique is to study how the magnetization of the structure is correlated to the stress loading changes.

Moreover, by means of the SQUID microscopy detection it is possible to obtain information about magnetization intensity due to the single slip band. In this way, it is possible to study where the mechanical stress produces main changes of the sample magnetization. An example is shown in figure 7(right) where a single scan, extracted by the magnetic field map, is reported. The line plot (*a*) represents the correlation between the normal component of the magnetic field gradient and two slip bands. The magnetic field intensity, measured as $\mathrm{d}B_z/\mathrm{d}x$, is different for each slip band. This

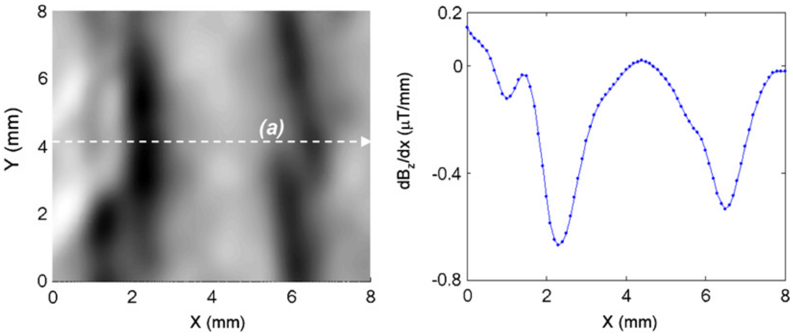

Figure 7. (Left) Magnetic field gradient $\mathrm{d}B_z/\mathrm{d}x$ corresponding to the slip bands area; (right) line scan (*a*) extracted from the map on the left.

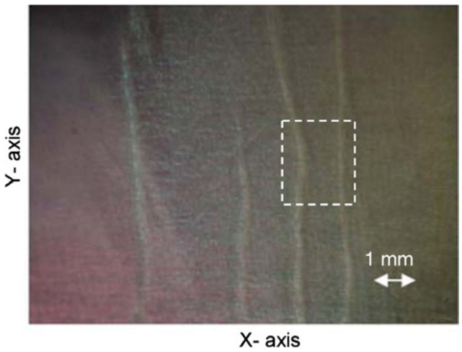

Figure 8. Optical image of the Fe 360 slip bands measured on sample 2.

means that the magnetization of the sample due to the plastic deformation is not uniform on the scale of a few millimetres across the specimen. Moreover, because the slip bands are directly correlated with the crack initiation, measuring the magnetization distribution it is possible to detect the mechanical behaviour of the structures until failure.

The SQUID microscope image could be compared with an optical image of the surface, as reported in figure 8. The SQUID microscope image reproduces the optical picture (white dot line) with the advantage that the magnetic field imaging gives information not only on the surface profile but mainly on the local magnetization of the sample.

5. Conclusions

The scanning SQUID microscopy technique has been used to investigate the magneto-mechanical effect present in structural steel, such as Fe 360.

The SSM represents one of the most sensitive techniques that could be applied to investigate and localize plastic deformation and crack grown initiation due to a tensile stress in structural steel.

The vertical component of the magnetic field has been detected and correlated to the variation of the dislocation density due to the applied mechanical stress. The capability of the SSM to detect signal due to magnetic slip bands, produced by plastic deformation, has been reported.

The magnetic imaging obtained by means of the scanning SQUID microscope demonstrates that the tensile stress creates a concentration of dislocations around the slot tip, where plastic deformation of the sample surface and crack initiation appear.

Moreover, on the dog-bone shaped specimen, it has been demonstrated that the spatial magnetic field gradient $\mathrm{d}B_z/\mathrm{d}x$ localizes the position of slip bands and measures the amplitude of the magnetic field correlated with them. This could be of great interest at research level and for the designer to obtain information about the severity of the fatigue stress and crack initiation in the structure.

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