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The influence of processing variables, on the extent of magnetically induced recovery in nickel

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Abstract. Doppler broadened positron annihilation spectroscopy has been used to investigate the effects of pulsed magnetic fields on the defect structure of heavily cold-worked nickel samples. The experimental variables investigated are processing temperature (−196 to 100 °C) and the relationship between the external field direction and sample texture. The key results are: (i) the maximum extent of magnetically induced recovery occurs at an intermediate temperature of around −80 °C, and (ii) the extent of magnetically induced recovery can be increased if the sample is subjected to multiple magnetic treatments conducted at different field/sample orientations.

1. Introduction

The interaction between magnetic fields and atomic-scale defects in ferromagnetic crystals has been under investigation for some time. The presence of dislocations in Ni has been shown to impede the motion of certain types of Bloch walls such that the coercive field strength is altered substantially [1]. It has also been demonstrated that pulsed magnetic fields can alter the defect structure and mechanical properties of Ni [2–9]. In this work, Doppler broadened positron annihilation spectroscopy has been used to investigate the influence of magnetic processing temperature (in the range −196 to 100 °C) on the extent of magnetically induced recovery in Ni. The influence of external field orientation with respect to sample texture has also been studied. Due to the competition between the thermal and magnetic contributions to the energy required for a dislocation to overcome obstacles in a deformed crystal, the maximum amount of magnetically induced recovery occurs at an intermediate processing temperature (about −80 °C). The interaction mechanism is believed to be associated with dislocation and Bloch wall stress fields. Since this interaction is significant only between certain types of dislocations and Bloch walls, the extent of magnetically induced recovery for any orientation of external field and sample texture is limited. If, however, the sample is rotated within the external field, new favourable combinations of dislocations and mobile Bloch walls occur and the extent of magnetic recovery increases.

2. Background

2.1. Positron annihilation spectroscopy

PAS is a non-destructive technique used to characterize atomic scale defects in materials. When a positron is injected into a sample it diffuses through the lattice until it annihilates

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with an electron to produce two γ-rays. If both particles were at rest, conservation of momentum and energy requires the two γ-rays to be emitted in opposite directions, each with an energy of 511 keV. When the annihilation event occurs at finite temperature, however, the net momentum of the electron/positron pair modifies the annihilation radiation characteristics. The γ-rays are no longer emitted anti-parallel to one another and their energies deviate from 511 keV. The technique that measures the energy deviations, ΔE, is known as *Doppler broadened* PAS (DBPAS). Due to the relative velocities of the positron and electron, the magnitude of ΔE is determined primarily by the electron momentum.

Positrons are attracted to, and often annihilate within, open volume defects such as vacancies, dislocations, and grain boundaries. Since the electron momentum distribution at a defect site is characteristic of that defect, the DBPAS lineshape is a fingerprint of the defect structure in the material. Thus, by monitoring changes in the DBPAS lineshape it is possible to track changes in defect types and relative concentrations. In this work, DBPAS spectra have been interpreted using the standard S parameter [10]. The S parameter is a measure of defect concentration that generally increases with increasing defect density.

2.2. Interaction between magnetic fields and defects in Ni

Previous work by a variety of researchers has demonstrated that external magnetic fields influence the mechanical and physical properties of metals [2–9]. Magnetic fields interact with atomic scale defects in ferromagnetic crystals in a variety of ways as described below.

Adjacent magnetic domains are separated by boundaries known as Bloch walls. Inside the wall the magnetization direction changes from one neighbouring domain to the other. Wall thickness is determined by two competing terms—the exchange energy and the anisotropy energy. Domain walls are described by the angle between the magnetization vectors in the neighbouring domains. Nickel, with its $\langle 111 \rangle$ easy magnetization directions, can have 180°, 110°, or 71° walls.

A magnetic field acts to increase the net magnetic moment in the direction of the field. This occurs either by the growth of favourably oriented domains via Bloch wall motion, or by dipole rotation within a domain. Magnetization of a ferromagnetic crystal also causes its length to change, inducing a strain. If adjacent domains have different dipole orientations there will be a strain mismatch across the Bloch wall. The elastic deformation necessary to accommodate this strain mismatch requires an increase in the energy of the system, known as the magnetostrictive energy.

Although the stress field associated with a domain wall is composed of contributions from several sources including exchange energy and magnetocrystalline anisotropy, it is due mainly to magnetostriction. Since adjacent domains with different dipole orientations experience different amounts of magnetostriction, and since the domains are not free to deform independently, microstresses are induced. This magnetically induced stress field can then interact with the stress fields associated with nearby dislocations. This hypothesis is supported by the observation that cold working makes the magnetization process more difficult since the resulting dislocations impede Bloch wall motion. As a result, cold-worked samples show a slower approach to saturation magnetization [1].

The magnitude of the interaction between dislocation and domain wall stress fields has been investigated [1, 11]. Seeger *et al* [12] considered the interaction between a $(\overline{1}\overline{1}2)$ 180° wall in Ni and a screw dislocation parallel to $[\overline{1}\overline{1}0]$. Their calculation, based on the Peach–Koehler equation, yields an induced shear stress of 1.3×10^7 N m^{-2}. This value is larger than the critical resolved shear stress for Ni which is $\sim 8 \times 10^6$ N m^{-2} at $-195\,^\circ$C and 7×10^6 N m^{-2} at $27\,^\circ$C [13]. Thus, it is reasonable to believe that during magnetization the force exerted on a dislocation by a moving domain wall is large enough to move the dislocation.

3. Experimental procedure

3.1. Doppler broadened position annihilation spectroscopy

The DBPAS system is composed of the following ORTEC components: a GEM-13180-P high-purity germanium detector powered by a 659 detector bias supply, a 672 spectroscopic amplifier and pile-up rejecter, a 444 gated biased amplifier, and a 919 multi-channel buffer. The energy dispersion for this system was ~ 48 eV/channel and each spectrum contained 201 channels for a total width of 511 ± 4.8 keV. Data collection was terminated when 20 000 counts were recorded in the peak channel resulting in ~ 1.3 million counts in each spectrum and data collection time of ~ 35 minutes per experiment.

Each energy spectrum was characterized by the standard S parameter defined as the ratio of the number of counts in a fixed central energy window to the total number of counts in the spectrum. The central energy window was 39 channels wide (511 ± 0.912 keV). The S parameters reported in this work represent the average values of at least five PAS experiments for each pair of samples. A typical value for the S parameters reported in this work is 0.5375 with a standard deviation of ~ 0.0003.

3.2. Sample preparation

The material investigated was polycrystalline Ni of 99.998% purity supplied by Material Research Corporation. The Curie temperature of Ni is 358 °C. The magnetocrystalline anisotropy of Ni results in $\langle 111\rangle$ being an easy direction of magnetization, $\langle 110\rangle$ intermediate and $\langle 100\rangle$ hard. The important types of domain walls in Ni are 70.53° walls lying in $\{100\}$ and $\{110\}$ planes, 109.47° walls lying in $\{100\}$, $\{110\}$ and $\{111\}$ planes and 180° walls lying in $\{110\}$ and $\{112\}$ planes [14, 15].

The Ni samples were cut from a cylindrical rod ~ 12 mm in diameter. The material was cold worked ~ 80% using the procedure described as process A in [9] resulting in the development of a $\{132\}\langle 112\rangle$ texture. The samples for PAS measurements were then cut to dimensions of ~ 10 mm × ~ 10 mm and had a final thickness of ~ 1.2 mm.

3.3. Magnetic treatment

The pulsed magnetic fields were generated by a Fluxatron model U-102 obtained from Innovex Corporation. This device consists of a magnetic coil that can provide several different field strengths. The magnetic treatments were performed at a series of temperatures in the range -196 to 100 °C.

The energy, emitted by the unit in a pulsed waveform, is absorbed by a specimen which is placed in a sliding tray in the centre of the coil. Each 'round' of energy output is fixed by a microprocessor within the Fluxatron to a duration of 42 seconds. The low field/high frequency setting was used which corresponds to a maximum field strength of ~ 6400 A m^{-1} with 630 pulses/round (i.e. 630 pulses in 42 s).

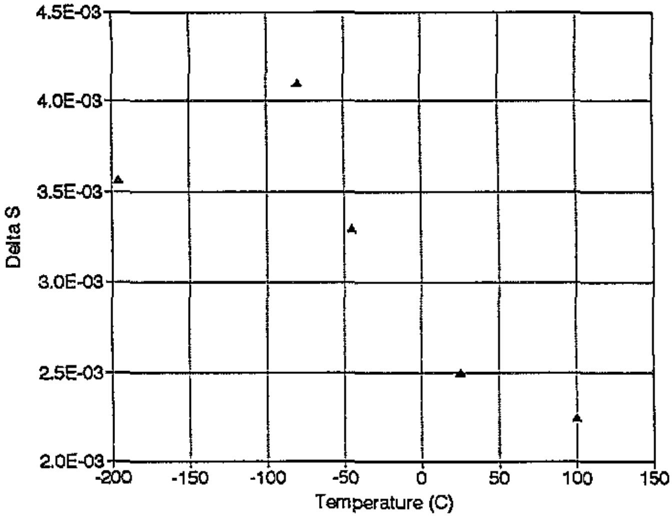

Figure 1. The magnitude of the magnetically induced change in the DBPAS S parameter, ΔS, as a function of magnetic processing temperature.

4. Results

4.1. Influence of temperature on the extent of magnetically induced recovery

In this phase of the project all samples were cold worked ~ 80% and then subjected to magnetic processing at temperatures in the range −196 to 100 °C. The samples were loaded in the Fluxatron so that the axis of the coil coincided with the rolling direction. Each sample pair was characterized by DBPAS before and after magnetic treatment. Since magnetic processing reduces the defect concentration in the samples it also reduces the observed value of S. In figure 1, the influence of magnetic processing temperature on the extent of magnetically induced recovery is shown in the form of ΔS versus temperature where ΔS is the difference between the S parameters before and after magnetic processing. The magnitude of ΔS is proportional to the extent of defect recovery. Note that the maximum amount of magnetically induced recovery occurs at an intermediate temperature of approximately −80 °C.

Figure 2 shows the same ΔS versus temperature data as in figure 1, but this time ΔS has been normalized by dividing each value by the maximum value (i.e. the value obtained at −80 °C). For comparison, figure 2 includes a reconstruction of the coercive field strength, H_c, data taken from reference [1]. These values were scaled such that they range from a maximum of 1.0 at approximately −100 °C to a minimum of 0.0 at 275 °C (the highest temperature for which data exists in [1]). The H_c data reflect the influence of temperature and dislocations generated during plastic deformation on the mobility of Bloch walls in Ni. The trends in ΔS and H_c with temperature are qualitatively similar. Also included in figure 2 is a series of points representing our simplified model for the temperature dependence of the thermal and magnetic contributions to the energy required for a dislocation to overcome obstacles in a deformed crystal. This model is described below.

4.2. Influence of field orientation and sample texture on the extent of magnetically induced recovery

In this phase of the project, a pair of samples was subjected to a second magnetic processing

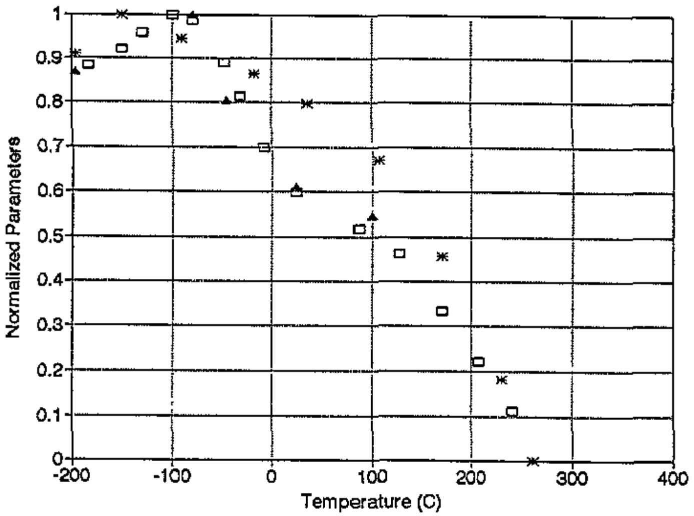

Figure 2. The variation of ΔS (filled triangles) and H_c (empty squares) with temperature. The theoretical points (asterisks) were generated using the model described in the text.

treatment in which the samples were loaded in the Fluxatron so that the axis of the coil was perpendicular to the rolling direction. The samples were treated at −196 °C in the initial and secondary treatments to isolate the influence of (external field)/(sample texture) on the extent of recovery. The results of this experiment are shown in figure 3. The horizontal axis is a listing of the various processing conditions while the vertical axis shows the extent of magnetic recovery as measured by ΔS. The ΔS value for the parallel treatment (coil axis parallel to rolling direction) is equivalent to the corresponding value reported in figure 1. The ΔS value for the perpendicular treatment (coil axis perpendicular to rolling direction) was obtained after the second processing operation.

As shown in figure 3, the initial 'parallel' magnetic processing operation resulted in a ΔS (reduction) of 0.017 and the 'perpendicular' treatment resulted in an additional reduction of ΔS of 0.014 for a total ΔS of 0.031. Thus, the second treatment produced an 80% increase in the extent of magnetically induced recovery.

5. Discussion

As shown in figure 2, ΔS and H_c display a similar dependence on magnetic processing temperature. In the case of ΔS, the local maximum is a result of the competition between the thermal and magnetic contributions to the energy necessary for a dislocation to overcome local obstacles in the lattice. The thermal contribution increases exponentially with increasing temperature while the magnetic contribution increases with decreasing temperature due to an increase in the strength of the exchange interaction. The result is that the maximum amount of magnetically induced recovery occurs at an intermediate temperature.

The theoretical curve shown in figure 2 was generated using a model similar to that used in [16] for explaining the temperature dependence of flow stress in a crystal. We assume a dislocation encounters a series of obstacles each of which exerts a force, K, on the dislocation as shown in figure 4. The magnitude of the interaction force is a function

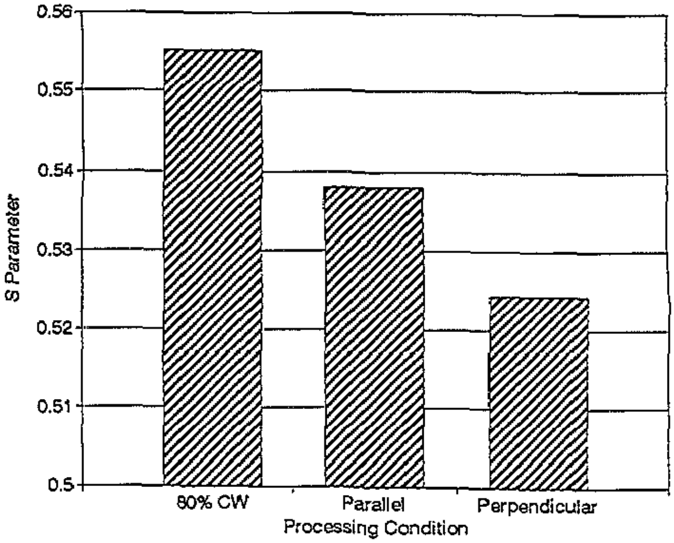

Figure 3. Influence of sample/field orientation on the extent of magnetically induced recovery as measured by the S parameter. The cold-worked sample was first treated with the magnetic field 'parallel' to the rolling direction and then treated with the field 'perpendicular' to the rolling direction.

of the distance, x, between the dislocation and the obstacle. If ΔF^* represents the area under the K–x curve and if the dislocation is subjected to an applied resolved shear stress τ^*, then

$$\Delta G^* = \Delta F^* - \tau^* V^* \tag{1}$$

where V^* is the activation volume for the process and ΔG^* is the (Gibbs) free energy of activation, or the thermal energy required for the dislocation to overcome the barrier.

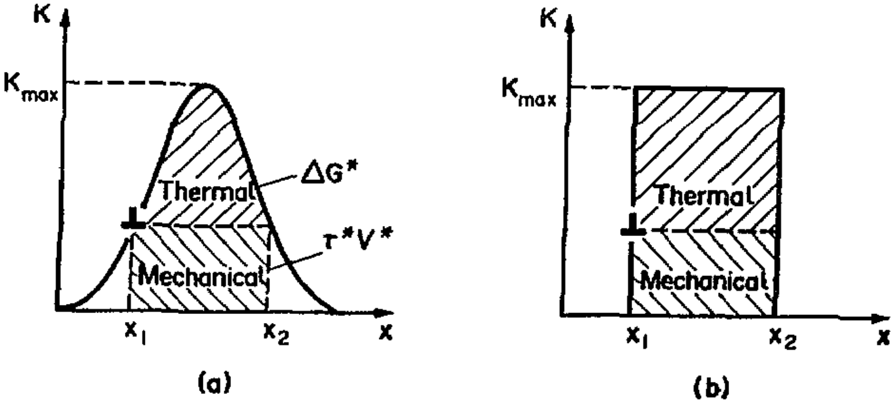

Figure 4. Profiles of resistance force, K, versus distance, x, for barriers opposing dislocation motion (adapted from reference [16]).

The probability of energy ΔG^* occurring by thermal fluctuations at temperature, T, is given by the Boltzmann factor $\exp(-\Delta G^*/kT)$. If the force versus distance curve shown in figure 4(a) is approximated using the one shown in figure 4(b), then:

$$\Delta G^* = \Delta F^*\{1 - [\tau^*(T)/\tau^*(0)]\} \tag{2}$$

where $\tau^*(T)$ is the flow stress at temperature T and $\tau^*(0)$ is the flow stress at 0 K. These equations can be algebraically manipulated to solve for the $\tau^*(T)$.

The temperature dependence of the magnetic contribution to dislocation motion can be modelled by replacing the quantity $\tau^*(t)$ in equation 3 with an equivalent magnetic 'energy' term $M^*(T)$. In this case, the probability, $P(T)$, of energy ΔG^* occurring by thermal fluctuations at temperature T is given by

$$P(T) = \exp(-\Delta G^*/kt) = \exp(-(\Delta F^*/kT)(1 - (M^*(T)/M^*(0)))). \tag{3}$$

Taking the logarithm of both sides of this equation, and absorbing the constant ΔF^* by relaxing the equality to a proportionality, yields

$$\ln(P(T)) \propto -((1 - (M^*(T)/M^*(0)))/kT). \tag{4}$$

The ratio $M^*(T)/M^*(0)$ represents the magnitude of the magnetic contribution to the motion of a dislocation normalized by the corresponding value at 0 K. We have chosen to use the relative saturation magnetization as a function of temperature, with experimental values obtained from reference [15], to estimate the ratio $M^*(T)/M^*(0)$ in equation 5. The theoretical curve shown in figure 2 is a normalized plot of this equation. It is interesting to note that this extremely simplified model corresponds reasonably well with both the ΔS and H_c data.

The H_c data, taken from [1], reflects the influence of dislocations on the mobility of Bloch walls as a function of temperature. In a sense it is the inverse problem—dislocations influencing Bloch wall motion rather than Bloch walls influencing dislocation motion. We believe, however, that both phenomena are controlled by the interaction between the stress fields of the Bloch walls and the dislocations.

Trauble [1] has explained the shape of the H_c versus T curve in heavily deformed Ni single crystals by modelling the coercive field strength as a function of the Bloch wall thickness, the shear modulus, the magnetostriction constants, and the spontaneous magnetization. The local maximum in the H_c data results from a change in the important magnetization mechanism from Bloch wall motion at low temperatures to dipole rotation at high temperatures. This analysis is not directly applicable to the polycrystalline samples examined in our present work, however, since it does not include the strong magnetostatic coupling between grains which is difficult to treat mathematically [1].

The data in figure 3 shows that the extent of magnetically induced recovery can be increased if the sample is subjected to a second series of magnetic treatments conducted at a different sample/field orientation. The magnitude of the force between a dislocation and a Bloch wall depends on the projections of the Burgers vector, $\boldsymbol{b}$, and tangent vector, $\boldsymbol{t}$, of the dislocation onto the plane of the Bloch wall. If $\boldsymbol{b}$ or $\boldsymbol{t}$ are perpendicular to the wall, then wall motion will have no effect on the dislocation. Additionally, only certain types of Bloch walls have long range stress fields capable of interacting with dislocations. In nickel, the important types of Bloch walls are (100) 71°, (110) 71°, (110) 109°, and (111) 109° [1].

Thus, for a fixed sample/coil geometry, only a limited number of appropriate Bloch walls will move in response to the external field and only a subset of the dislocations will be favourably oriented such that significant interaction can occur. By changing the sample/coil alignment, however, new combinations of interacting Bloch walls and dislocations are obtained and additional magnetic recovery occurs.

Despite the use of the optimum processing temperature and two sample/field orientations the maximum extent of magnetically induced recovery is still only ~ 50% of that obtained by thermal annealing. The reason may be that the magnetic field does not interact strongly with point defects [9]. Thus, during magnetic processing and significant reduction in point defect concentration occurs, and perhaps more importantly, the absence of dislocation climb limits dislocation mobility.

6. Conclusions

Doppler broadened positron annihilation spectroscopy has been used to investigate the effects of processing temperature and the field/sample orientation on the extent of magnetically induced recovery in nickel. The maximum amount of magnetically induced recovery occurs at an intermediate temperature of −80 °C. It is believed that this is a result of the competition between the thermal and magnetic contributions to the energy necessary for a dislocation to overcome local obstacles in the lattice. The thermal contribution increases exponentially with increasing temperature while the magnetic contribution increases with decreasing temperature due to an increase in the strength of the exchange interaction.

The extent of magnetically induced recovery can be increased by subjecting the sample to multiple magnetic treatments at different orientations. Although there are only a limited number of favourable Bloch wall/dislocation combinations for any given applied field direction, by changing the sample/coil alignment new combinations are obtained and additional recovery occurs.

Acknowledgments

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References

[1] Trauble H 1967 *Magnetism and Metallurgy* vol 2, ed A E Berkowitz and E Kneller (New York: Academic) pp 622–45
[2] Cullity B D and Allen C W 1965 *Acta Metall.* **13** 933
[3] Hayashi S, Takahashi S and Yamamoto M 1971 *J. Phys. Soc. Japan* **30** 2
[4] Collins J D 1976 *US Patent* 3,963,533
[5] Pavlov V A, Pereturina I A and Pecherkina N L 1980 *Phys. Status Solidi* a **57** 449–56
[6] Jablonowski J 1987 *Pulsed Magnetism Boosts Tool Life* (American Machinist & Automated Manufacturing) pp 80–2
[7] Hochman R F, Tselesin N and Drits V 1988 *Adv. Mater. Proc.* **134** 36
[8] Su Y Y, Hochman R F and Schaffer J P 1990 *J. Phys.: Condens. Matter* **2** 3629–42
[9] Vaidyanathan R, Schaffer J P and Thanaboonsombut B 1993 *J. Phys.: Condens. Matter* **5** 4563–72
[10] MacKenzie I K, Eady J A, Gingerich R R 1970 *Phys. Lett.* **33A** 179
[11] Vicena F 1954 *Czech. J. Phys.* **5** 480
[12] Seeger A and Kronmuller H 1987 *Mater. Sci. Forum* **15–18** 65–70
[13] Haasen P 1958 *Phil. Mag.* **3** 384
[14] Chen C W 1986 *Magnetism and Metallurgy of Soft Magnetic Materials* (New York: Dover)
[15] Cullity B D 1972 *Introduction to Magnetic Materials* (Reading, MA: Addison-Wesley)
[16] Hull D and Bacon D J 1984 *Introduction to Dislocations* (Oxford: Pergamon)