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Analysis of cold rolled steels of different reduction ratio using the magnetic Barkhausen noise technique

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Magnetic Barkhausen Noise (MBN) was used to evaluate the magnetic behaviour of nuclear reactor pressure vessel steel specimens cold rolled to reduction ratios between 0% and 60%. Measurements performed at increasing reduction ratios revealed variations in the angular dependence of a parameter termed '$\mathrm{MBN}_{\mathrm{energy}}$', concurrent with modifications in the shape of the pulse height distribution curves. The angular preference of $\mathrm{MBN}_{\mathrm{energy}}$ present prior to cold rolling was destroyed at intermediate reduction ratios (≈25%), and restored with further reduction (≈40%). Along the rolling direction, the number of large voltage pulses was reduced at reduction ratios of ≈25%, increasing again at ≈60% reduction ratio. Results were attributed to competing effects between crystallographic texture, microscopic and macroscopic residual stresses. *© 2001 Kluwer Academic Publishers*

1. Introduction

'Magnetic anisotropy' implies that magnetic properties depend on the direction in which they are measured. The design of some ferromagnetic materials of commercial importance depends on their magnetic anisotropy [1]. Magnetic anisotropy changes have been observed [2] in some ferromagnetic alloys after cold rolling. Investigations performed on iron-aluminum [3], and nickel-cobalt [4] alloys using a torque magnetometer revealed variations in torque values along one direction in the iron-aluminum alloys, and along two directions in nickel-cobalt. The magnetic anisotropy observed in these studies has been attributed to the rolling process, and has been termed 'roll magnetic anisotropy' [2–4]. The exact mechanism responsible for roll magnetic anisotropy has not been discussed, however. In an attempt to further understand the origins of magnetic anisotropy, the present study uses the magnetic Barkhausen noise (MBN) technique to examine relative changes in magnetic anisotropy between different cold rolling stages of nuclear reactor pressure vessel steel specimens.

2. Theoretical background

Under the influence of an applied time-varying magnetic field, a ferromagnetic material produces voltage signals in a surface-mounted pick-up coil. These voltage signals are associated with MBN, which occurs primarily in the central region of the magnetic hysteresis curve where domain wall motion is the dominant magnetization mechanism [1]. The irreversible motion of domain walls across local pinning sites such as dislocation tangles gives rise to MBN [1]. Increases in dislocation density with plastic deformation lead to selective increases in the domain wall energy gradient at pinning sites, resulting in a larger MBN response [5].

In the present study, the MBN signal is characterized by a parameter termed '$\mathrm{MBN}_{\mathrm{energy}}$' [6] defined as the integral of the square of the MBN voltage over the signal envelope. The magnetic anisotropy in the material is determined by conducting angular MBN measurements, where the MBN magnet is rotated about a single axis with measurements taken every 10°. Typically the data is displayed in polar form with $\mathrm{MBN}_{\mathrm{energy}}$ plotted as a function of angle. A curve described by [7]

$$\mathrm{MBN}_{\mathrm{energy}} = \alpha \cos^2(\theta - \phi) + \beta \qquad (1)$$

is fit to the data points. The fitting parameter β is associated with isotropically oriented domains, (essentially the background signal) while α corresponds with domains responsible for a magnetic easy axis [7]. The angle θ denotes the direction of the applied magnetic field with respect to the angle ϕ of the easy axis of magnetization. An elongation of the $\mathrm{MBN}_{\mathrm{energy}}$ polar plot in a specific direction implies an easy axis of magnetization, and the size of the α parameter gives an indication of magnetic anisotropy.

Severe deformation of steel, such as occurs during cold rolling, leads to a $\{100\}\langle 110\rangle$ texture development which generally can be detected after about 20% to 30% reduction [8]. Appreciable scatter about this 'ideal' texture still exists at this deformation stage. At about 80% to 90% reduction the $\{100\}\langle 110\rangle$ texture development is essentially complete [8].

TABLE I Chemical composition (in wt%) of ASTM A 508 class 3 nuclear reactor pressure vessel steel

C	Si	Mn	P	S	Ni	Cr	Mo	Al	Cu	V	Co
0.18	0.03	1.48	0.004	0.003	0.91	0.21	0.54	0.003	0.045	0.003	0.004

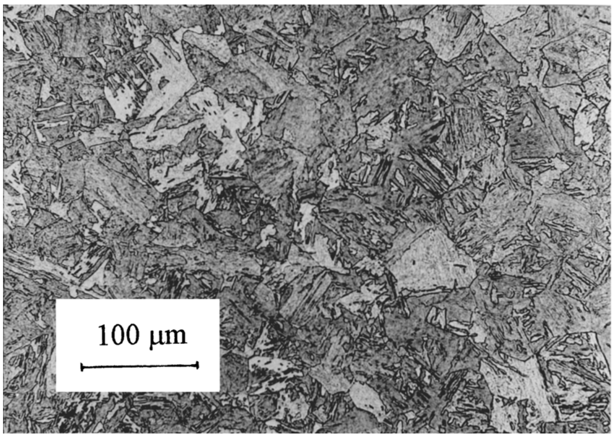


Figure 1 Photomicrograph of cold rolled specimen of 0% reduction ratio.

Residual stresses are internal elastic stresses ‘trapped’ in the material as a result of inhomogeneous deformation. They may be macroscopic across a sample, or may be microscopic, building up at grain boundaries or even between crystallographic planes which deform differently in response to an applied stress. MBN has been used successfully for residual stress evaluation in steels [9], although the MBN technique is still in its early stages of development.

3. Experimental technique

The samples investigated were of ASTM A 508 class 3 nuclear reactor pressure vessel steel. The processing treatments applied to the material were: austenizing at 1143°K for 4.5 h, water quenching and then tempering at 928°K for 9 h, followed by air cooling. Heat treatment for stress relief was performed at 880°K for 48 h. The chemical composition is given in Table I.

Samples for microstructural examination were prepared by grinding on SiC impregnated paper followed by polishing using successively finer diamond grit sizes. To reveal the microstructure, a 2% Nital etch was used. The microstructure, shown in Fig. 1, consists of a combination of lath and equiaxed ferrite grains.

The specimens were machined to plates of 60 mm length, 20 mm width and thickness varying between 3.0 and 7.5 mm. These specimens were cold rolled to reduction ratios of 0, 1.6, 3.2, 6.3, 9.1, 14.3, 19, 25, 30.2, 34.8, 40, 45.5, 50, 53.9, and 60%.

The measurement equipment and procedure used to acquire an MBN signal are described elsewhere [6].

4. Results

Fig. 2 shows, for a few selected samples, the changes in magnetic easy axis of the cold-rolled steels as a function of reduction ratio. Fig. 2a shows an axial magnetic easy axis in the ‘as received sample’ (at zero reduction ratio). With some cold rolling this initial MBN$_{\text{energy}}$ orientation preference appears to have been destroyed (Fig. 2b). Additional plastic deformation re-creates a direction of easy magnetization, as shown in Fig. 2c, however shifted towards the transverse direction. Still higher plastic deformation results in a very pronounced magnetic easy axis, and a simultaneous rotation of the latter toward the rolling direction (Fig. 2d).

Fig. 3 shows the changes in the α and β parameters with reduction ratio for all of the samples studied. In the undeformed state and slightly above, the parameter α is slightly higher than β, consistent with the preferred angular orientation observed in the undeformed state and small plastic deformation. At intermediate reduction ratios, however, β becomes slightly dominant over α, which coincides with the loss of the easy axis. Further increase in reduction ratio creates a significant rise in α parameter values, corresponding to the strong easy axis at the highest deformation levels.

In addition to the angular MBN experiments, measurements were also done with the MBN detector oriented parallel to the rolling direction and scanned across a sample surface. Average MBN$_{\text{energy}}$ values obtained from these scans (Fig. 4) show a drop at intermediate stages of cold rolling, occuring simultaneously with the loss of anisotropy revealed in Fig. 2b. Higher reduction ratios produce an increase in MBN$_{\text{energy}}$, similar to the one observed for the parameter α.

Pulse height distributions examined for selected samples for detector orientations of 0°, 90°, and 45° with respect to the rolling direction (Fig. 5) reveal a few notable aspects. For the detector oriented parallel to the rolling axis, distribution curves become taller and narrower during intermediate stages of cold rolling, but broaden again at high reduction ratios, as shown in Fig. 5a. A different trend is revealed in the transverse direction (Fig. 5b) where the ‘tail’ of the distribution curve (larger voltage pulses) increases slightly at intermediate reduction ratios, but contracts significantly during the last stages of cold rolling, with a considerable rise in the low voltage pulses. At 45° to the rolling direction little difference is noted between reduction ratios (Fig. 5c). Results illustrated in Fig. 6 for a single reduction ratio of 60% reveal small orientation dependence for 0° to 45°, however, at 90° a considerable increase in the number of low voltage pulses is observed.

5. Discussion

The metallurgical changes accompanying cold rolling are complex, and a number of different factors are likely to influence the magnetic anisotropy of the sample–crystallographic texture development, the formation of a significant (and possibly anisotropic) dislocation structure, and residual stresses on both a micro and a macro scale. The easy axis observed in the unrolled state

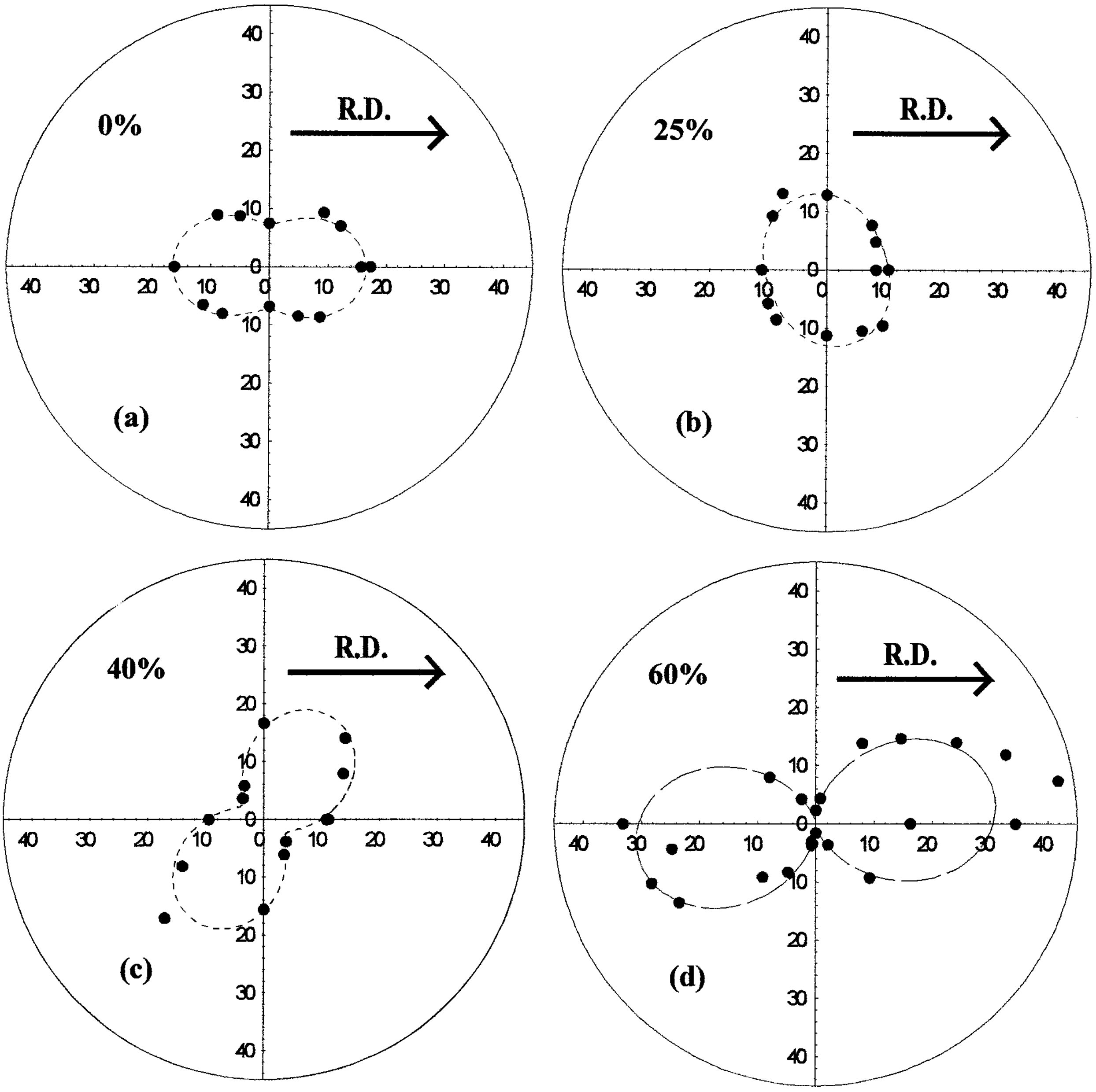


Figure 2 Selected polar plots of angular $\mathrm{MBN_{energy}}$ for reduction ratios of (a) 0%, (b) 25%, (c) 40%, and (d) 60%. A curve described by equation (1) is fit to the data.

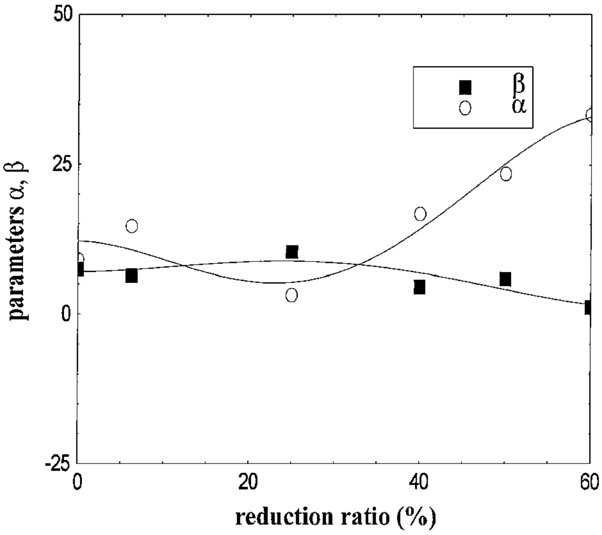


Figure 3 Variation with reduction ratio of parameters α and β of equation (1). Solid lines are only for guidance.

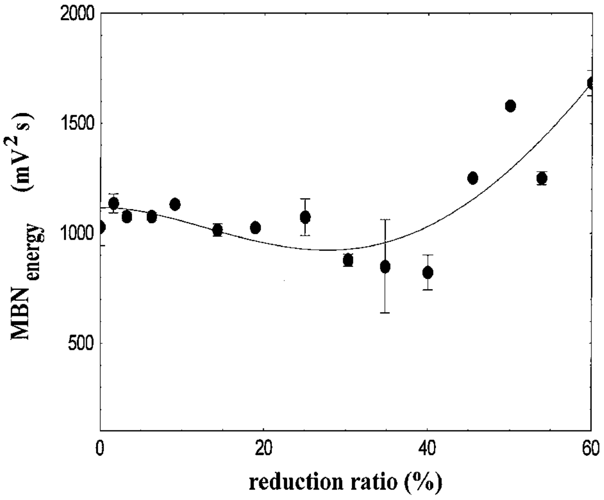
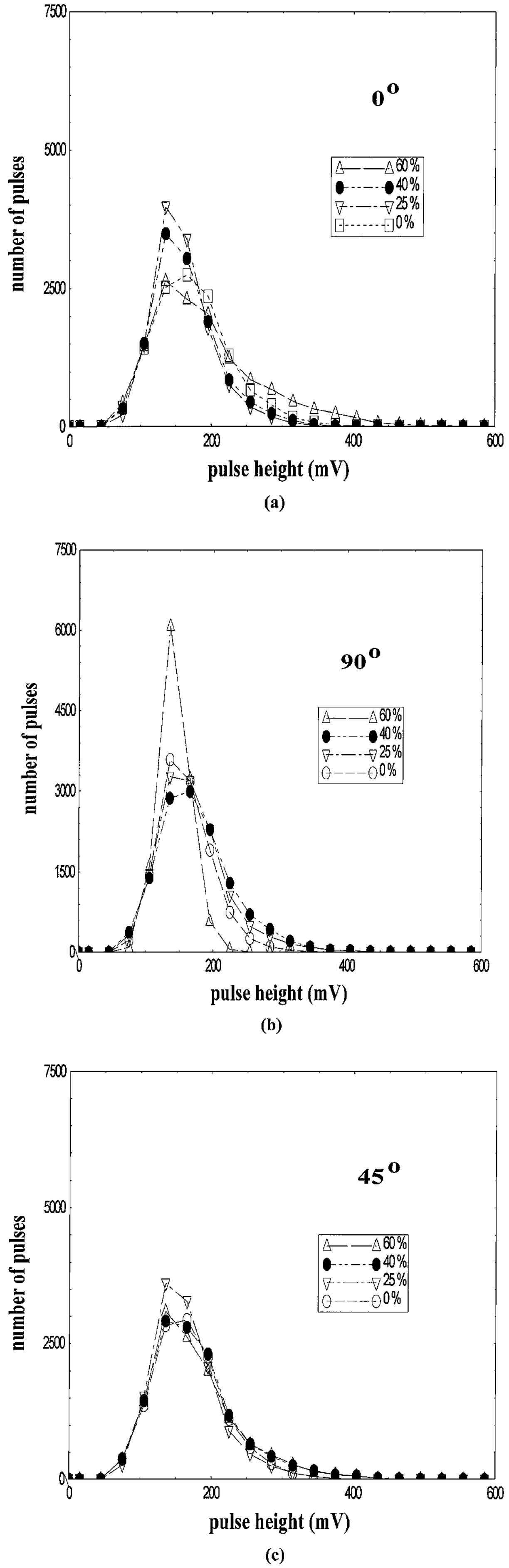


Figure 4 Variation with reduction ratio of $\mathrm{MBN_{energy}}$. Detector oriented parallel to the rolling direction. Connecting line is only for guidance. Error bars represent standard deviations.



(a)

(b)

(c)

Figure 5 Pulse height distributions for cold rolled specimens of selected reduction ratios. Measurements made with detector oriented (a) parallel, (b) transverse, and (c) at 45° to the rolling direction.

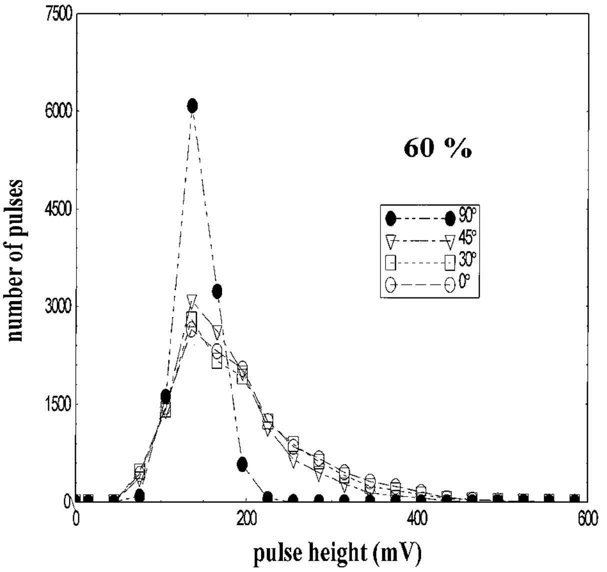


Figure 6 Pulse height distributions for cold rolled specimen of 60% reduction ratio for selected detector orientations.

(Fig. 2a) is likely a result of crystallographic texture, since considerable effort has been taken in processing to ensure that residual stresses are relieved (see experimental section). The initial rolling stages remove this magnetic easy axis as the initial crystallographic texture is destroyed (Fig. 2b).

The reappearance at higher reduction ratios of an inclined magnetic easy axis (Fig. 2c) which reorients into a strong axial easy axis (Fig. 2d), suggests that competing factors may be influencing the magnetic anisotropy. Macroscopic residual stresses are generally axially compressive at the surface of a cold rolled sample [8] which should encourage an easy axis transverse to the rolling direction [7]. This may be responsible for the inclination towards 90° at intermediate reduction ratios (Fig. 2c). The pulse height distributions at 90°, Fig. 5b, support this, since initially the number of large pulses (associated with large $\mathrm{MBN}_{\mathrm{energy}}$ values) increases.

With further rolling the influence of the compressive macroscopic residual stress appears to diminish, and a strong axial easy axis develops (Fig. 2d). At this point the reason for this axial easy axis has not yet been determined. It is unlikely to be crystallographic texture since the rolling texture direction is ⟨110⟩, yet magnetic texture is associated with the ⟨100⟩ direction. We speculate that at high reduction ratios the magnetic easy axis may result from an anisotropic distribution of dislocation tangles acting as domain wall pinning sites. Conversely it may also be associated with microscopic, or *intergranular* residual stresses [10]. These are residual stresses which accumulate in certain crystallographic directions as a material is plastically deformed. For example, the work of Pang *et al.* [10] showed that moderate (13%) tensile plastic deformation can create very large tensile residual strain in the ⟨100⟩ direction with little residual strain accumulation in the other crystallographic directions. Our work is continuing to establish the exact mechanism responsible for the strong axial easy axis which develops at these high reduction ratios.

6. Conclusion

Changes in magnetic anistropy of cold rolled nuclear reactor pressure vessel steel specimens were investigated using magnetic Barkhausen noise. An initial axial magnetic easy axis, likely resulting from preferred crystallographic texture, is destroyed early in the rolling process. It is replaced at intermediate reduction ratios by a tendency towards a transverse magnetic easy axis resulting from macroscopic axial compressive stresses developed at the sample surface during rolling. This effect competes with a further, as yet unknown, effect that finally dominates at very high reduction ratios. We speculate that this unknown effect is associated either with anisotropic dislocation-based pinning sites or intergranular residual stresses.

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