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# Influence of stress and stress-flash annealing on the magnetic properties of amorphous $\text{Fe}_{77.5}\text{Si}_{7.5}\text{B}_{15}$ wire

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The influence of the tensile stress on the flux reversal phenomena in as-quenched state of  $\text{Fe}_{77.5}\text{Si}_{7.5}\text{B}_{15}$  is investigated. A large and stable Barkhausen jump is observed at a field  $H^*$ , called the switching field. The switching field  $H^*$  passes through a minimum as tensile stress increases and the jump in magnetization  $M^*$  at  $H^*$  grows with stress. It is observed that  $H^*$  of the sample can be modified by flash annealing under stress. The results on magnetic properties like coercive field, squareness ratio, power loss of as-quenched and stress-annealed samples are presented. The stress annealing induces elastic and plastic components in anisotropy.

## INTRODUCTION

The magnetic properties like initial susceptibility,  $M$ - $H$  behavior, coercivity, etc., of amorphous alloys depend very much on the internal stress generated during the preparation. The nature, magnitude, and distribution of frozen stress in the amorphous state strongly depend on the process of rapid quenching and the morphology of the alloy. The differences in magnetic behavior of magnetostrictive alloys in the form of ribbons produced by melt spinning<sup>1</sup> and wires produced by in-water quenching<sup>2</sup> are due to different internal stresses.<sup>3</sup> After relaxing the internal stress by annealing for long durations of time similar magnetic properties are observed<sup>4</sup> for wire- and ribbon-shaped samples. This suggests that the magnetoelastic coupling in the presence of internal stress plays a predominant role in determining the magnetic properties of amorphous alloys. Among all the differences in magnetic behavior between wire and ribbon shaped samples the most striking feature is the low-field magnetic behavior. At low field, instead of any minor loop as generally observed in a magnetic system, a re-entrant flux reversal resulting in a large and stable Barkhausen jump occurs in magnetostrictive amorphous wire. The flux reversal appears when the applied field crosses a critical value—the switching field  $H^*$ . This switching characteristic makes the amorphous wire suitable as a pulse-generating sensor element for applications such as rotary encoder, noncontact switches, security sensor, etc.<sup>5,6</sup>

The model which describes the flux reversal at  $H^*$  assumes the existence of an axial domain within the inner core and a closure domain at the outer layer along with zig-zag walls at the surfaces.<sup>7,8</sup> The magnetization in the

axial domain is oriented parallel to the axis of the wire, whereas that in the closure domain is normal to the axis. The existence of tensile longitudinal stress within the inner core and radial stress within the outer region and finite magnetostriction are considered to be the reason for such complicated domain structure. In this model the re-entrant flux reversal is due to reversal of magnetization in the inner-core domain. This domain model is also used to interpret the magnetoresistance results on amorphous wire.<sup>9</sup> The large Barkhausen jump disappears when the outer region is removed by etching.<sup>10</sup> This underlines the importance of stress distribution (existence of radial and longitudinal stress) in the phenomena of flux reversal and other magnetic behavior of the wire.

Therefore, the study of flux-reversal behavior by changing the stress distribution can provide much insight into the phenomenon. It has been observed during ac magnetization measurements<sup>4</sup> that the field  $H^*$  changes very much in the presence of external tensile stress. So the switching field  $H^*$  can be controlled by introducing stress-induced anisotropy. This phenomenon has been studied here in  $\text{Fe}_{77.5}\text{Si}_{7.5}\text{B}_{15}$  wire which is flash annealed under external tensile stress. The switching field  $H^*$  of the annealed sample passes through a minimum as the magnitude of external stress applied during flash annealing increases. For an as-quenched sample, similar minimum is observed as a function of external tensile stress applied during the measurement of  $H^*$ . But the magnitude of  $H^*$  in an annealed sample is found to be much smaller than that in an as-quenched sample for a corresponding external stress. The results on the other magnetic properties like magnetization  $M^*$  at  $H^*$ , coercivity, power loss, etc., in as-quenched and annealed samples are also presented.

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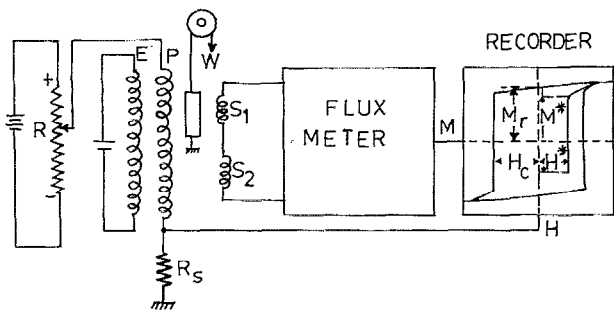


FIG. 1. Schematic diagram of the experimental setup together with the different parameters of the hysteresis loop.  $P$  is the primary,  $S_1$  and  $S_2$  are two secondaries and  $E$  is earth-field compensating coil.

## EXPERIMENT

Amorphous wire having a nominal composition of  $\text{Fe}_{77.5}\text{Si}_{7.5}\text{B}_{15}$  and produced by in-water quenching is used here. The sample of length  $\approx 7$  cm is placed within one of two identical secondary coils connected in series opposition. The secondaries are placed in a uniform magnetic field which can be varied continuously in forward and reverse directions. The induced voltage from the secondaries is integrated by an integrator (Walker Scientific Inc. MF-3D). The integrated voltage which is proportional to the magnetization is measured at different fields and is also recorded by an  $x$ - $y$  recorder. The earth's field is compensated by another static field. In Fig. 1, the schematic diagram of the setup and a hysteresis loop is shown. Here  $M^*$  is the discontinuous jump in magnetization at  $H^*$ . The coercive field ( $H_c$ ) and remanence ( $M_r$ ) are determined when the field is decreased from a large value where technical saturation has been observed. The power loss is obtained from the area of the hysteresis loop.

The flash annealing<sup>11</sup> is done by sending a current pulse through the wire in the presence of tensile stress. The current amplitude of 350 mA and 3 s duration is used, and higher amplitudes of current have been found to destroy the flux-reversal characteristic. The tensile stress is applied by loading one end of the wire while the other end is clamped. A separate piece of wire is taken for each annealing. The measurements on the stress-annealed sample have been made without any external stress and the value of stress given in the results of each annealed sample is the value of stress applied during annealing.

## RESULTS

The stress dependence of  $H^*$  in the as-quenched state of the wire-shaped amorphous alloy  $\text{Fe}_{77.5}\text{Si}_{7.5}\text{B}_{15}$  is given in Fig. 2. An almost linear decrease of  $H^*$  is observed up to  $\sigma = 260$  MPa beyond which it increases more steeply. For stress-annealed samples the nature of the variation of  $H^*$  with stress applied during annealing is similar to the as-quenched sample under external stress. The magnitude of  $H^*$  in this case is much smaller than that in the as-quenched sample for a corresponding external stress. This shows the usefulness of stress annealing in varying the

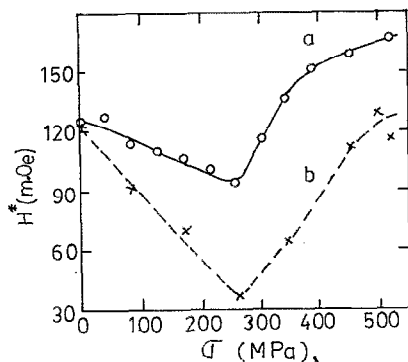


FIG. 2. Plot of switching field ( $H^*$ ) against the stress ( $\sigma$ ). Curve  $a$  for as-quenched sample under applied tensile stress ( $\sigma$ ) and curve  $b$  for sample annealed under different tensile stress ( $\sigma$ ).

magnitude of  $H^*$  which is desirable for application purpose. Therefore, the magnitude of critical field  $H^*$  can suitably be varied by an appropriate combination of stress and heat pulse during annealing. In order to see the usefulness of these annealed wire as pulse-generating sensor-element four annealed wires having different values of  $H^*$  (36, 64, 92, and 123 mOe) and of nearly identical length are put together and total change in magnetization of these wires due to Barkhausen jump is measured as a function of dc field  $H$ . The first discontinuous change in magnetization ( $M_T$ ) is observed around  $H \approx 36$  mOe but other jumps appear at higher fields compared to their respective values of  $H^*$  (Fig. 3). The magnetization reversal appearing at higher field is due to the demagnetizing field produced by wire which is magnetized at lower field. Although typical values of demagnetization factor lies around  $(2-5) \times 10^{-6}$  the demagnetization fields are in mOe range due to large values of magnetization ( $\sim 12-15$  kG).

The discontinuous jump of magnetizing  $M^*$  occurring at  $H^*$  shows a shallow minimum at low stress but increases linearly for higher stresses up to  $\sigma = 525$  MPa (Fig. 4) for the as-quenched state. For annealed samples

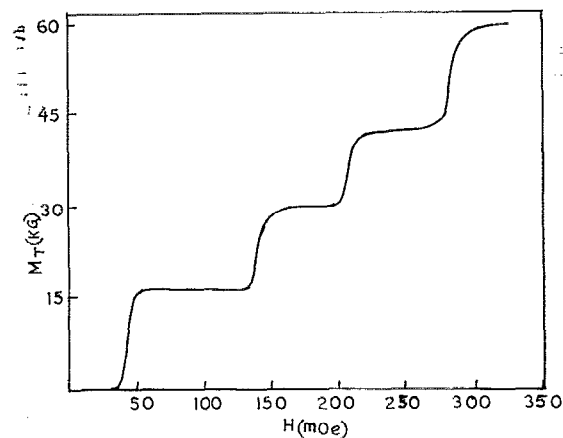


FIG. 3. Total change of magnetization ( $M_T$ ) with field  $H$  when four pieces of wire of different switching field ( $H^*$ ) are kept together. The values of  $H^*$  of four individual wires are 36, 63, 92, and 123 mOe.

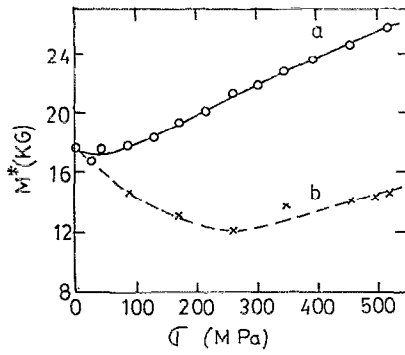


FIG. 4. Dependence of discontinuous jump in magnetization ( $M^*$ ) of the as-quenched sample (curve *a*) with stress  $\sigma$  and of annealed sample (curve *b*) with stress present during annealing.

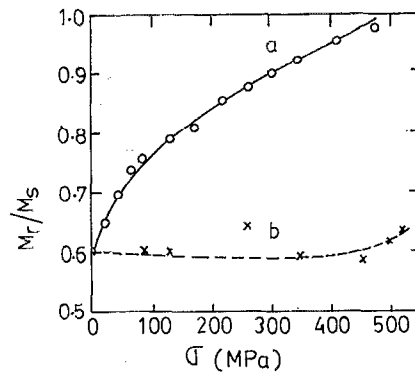


FIG. 6. Plot of squareness ratio ( $M_r/M_s$ ) of the as-quenched sample (*a*) at different tensile stress  $\sigma$  and of annealed sample (*b*) annealed at different stress  $\sigma$ .

the minimum appears at higher stresses (Fig. 4). The variation of coercive field  $H_c$  with stress, as shown in Fig. 5 is more complex. It exhibits a maximum at low stress and broad minimum at higher stress for both unannealed and annealed samples. The position of both the minimum and the maximum in the annealed case shifts towards a higher stress value compared to those of the as-quenched one. Fig. 6 represents the effect of tensile stress on the squareness ratio ( $M_r/M_s$ ). In the as-quenched state (Fig. 6) the remanence increases with stress and tends to saturate at higher stress. After stress annealing there is almost no change on the squareness ratio except for a slight increase in  $M_r$  at higher stress-annealed wires. Figure 7 represents the stress behavior of power loss determined from the area of the hysteresis loop. In the as-quenched sample (curve *a*) power loss increases for low values of stresses and shows a maximum at 60 MPa. It then starts decreasing giving a minimum around 200 MPa. After  $\sigma = 200$  MPa the power loss starts increasing rapidly. For the annealed case (curve *b*) the extent of the variation in power loss is reduced, the maximum is broadened and the minimum appears at higher stress value.

## DISCUSSIONS

Amorphous wires produced by in-water quenching technique have a unique distribution of internal stress. When the molten alloy hits the water the outer shell solidifies first and then the inner core solidifies and shrinks. Due to this differential cooling process a radial tension is created which increases towards the center of the wire resulting in an axial tensile stress at the inner region. Thus the internal stress distribution produces two distinct regions in the amorphous wires—one is an inner core where a tensile stress exists and the other is an outer core where a radial directional stress is present. The transition from longitudinal to transverse stress distribution is expected to occur gradually over a few atomic layers. The volume of the inner and outer core regions depends very much on the magnetoelastic coupling of the sample. These two internal stresses are long range in character and play a predominant role in determining the domain structure. For a positive magnetostrictive system like  $\text{Fe}_{77.5}\text{Si}_{7.5}\text{B}_{15}$  the axial tensile stress produces an axial domain (inner core) whose magnetization orients parallel to the axis. Similarly, the radial

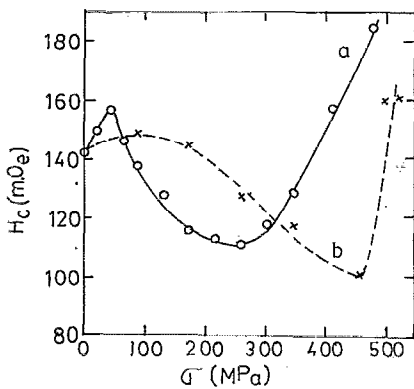


FIG. 5. Variation of coercivity ( $H_c$ ) of an as-quenched sample (curve *a*) at different stress  $\sigma$  and of annealed samples annealed at different stress  $\sigma$ .

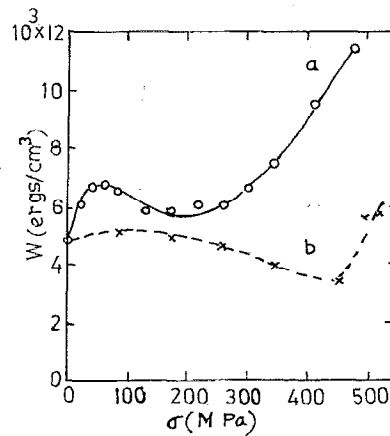


FIG. 7. Dependence of power loss  $w$  of the as-quenched sample (*a*) at different tensile stress  $\sigma$  while curve *b* is for the corresponding stress-annealed samples.

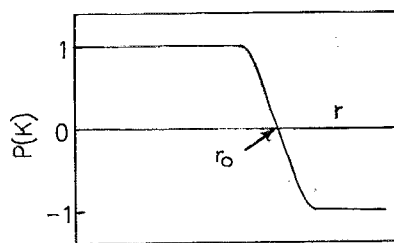


FIG. 8. Simplified picture for the magnetoelastic anisotropy distribution  $P(K)$  in amorphous wire along the radius  $r$ . Positive and negative values correspond to anisotropies with easy axis parallel or perpendicular to the ribbon axis, respectively.

stress produces a domain structure where  $M$  is perpendicular to the axis. To a first approximation, the distribution  $P(K)$  of axial anisotropy energy,  $K$ , originating from the coupling of the internal stress with the magnetization can be considered as shown in Fig. 8 and can be written in a simplest form as

$$P(K) = +1 \quad \text{for } r < r_0,$$

$$= -1 \quad \text{for } r > r_0,$$

where  $r$  is the distance from the center of the wire and  $r_0$  is the distance where the anisotropy energy changes its direction. The values of  $r_0$  depends on the magnetostriction constant and the stress distribution. In the case of low magnetostrictive wire the value of  $r_0$  is not distinguishable and hence it behaves like a ribbon.<sup>4,8</sup> When the tensile stress is applied to a positive magnetostrictive sample the stress distribution shifts to a higher stress level resulting in an increase in the value of  $r_0$ . These two regions have been studied by measuring the transverse and longitudinal magnetoresistance and a "self-similar expansion" model has been proposed by Makino *et al.*<sup>9</sup> The large Barkhausen jump is observed as the flux reversal occurs in the magnetized state. Using the Matteucci effect<sup>12</sup> the flux-reversal mechanism has been studied by Humphrey *et al.*<sup>8</sup> He observed that the re-entrant reversal proceeds in three steps. In the first step the nucleation starts at the two ends of the wire, grows by a constant flux change and finally there is a fast reversal as the two domain walls meet and collapse. The inhomogeneous freezing along the direction of the wire may produce some short-range stress centers which can be either tensile or compressive in nature. These short-range stresses are the pinning centers for the domain walls.<sup>13</sup> With the application of the applied tensile stress the effect of stress centers may reduce and the domain wall movement will be much easier. This explains the initial soft magnetic nature of the stress dependent properties of the as-quenched sample. The slow variation of  $M^*$  at low stress also depicts the presence of a random distribution of internal stress. It is not quite understood why in high-field measurements of coercivity and power loss, hard magnetic behavior has been observed for very low stress values. After this initial disparity between the high- and low-field behavior the stress-dependent magnetic properties are the same for both field regions.

When the applied stress is increased, the volume of the inner core increases at the expense of the outer core volume. This is incorporated in the increase of  $M^*$  or  $M_s$  value with the applied stress. Thus increase of the axial anisotropy region together with the reduction of short-range stress centers result in a monodomain structure. At higher value of stress the domains deform both in their shape and size and the distortion resists the easy movement of the domains. This consequently makes the system magnetically hard.

It has been found that the annealing of the wire for long durations of time (not long enough to destroy the amorphous nature) destroys the switching behavior.<sup>4,5</sup> This is due to the removal of the radial stress from the outer region of the wire. Flash annealing has been adopted to heat up the sample for short durations so that the nature of the internal stress distribution is not radically disturbed. It is observed from the behavior of  $H^*$  with stress applied during flash annealing that such treatment introduces frozen stress within the wire. At the same time some of the radial stress has been annealed out during the treatment. This is borne out from the fact that  $H^*$  in an annealed sample is reduced compared to that in an as-quenched sample at the corresponding stress. The effects of applied stress on the magnetic properties can be made permanent if the longitudinal stress can be "frozen in" inside the wire. The study of stress-annealed amorphous ribbon reveals that the fraction of tensile stress applied during annealing remains within the sample as a frozen in condition even when the applied stress is withdrawn.<sup>14</sup> By stress annealing, the switching field ( $H^*$ ) can suitably be controlled. This demonstrates the feasibility of using the amorphous wire for pulse-generating sensor elements where there is a continuous change of dc field.

## CONCLUSION

Stress and stress-annealing dependence of magnetic properties have been studied for amorphous  $\text{Fe}_{77.5}\text{Si}_{7.5}\text{B}_{15}$  wire produced by in-water quenching method. The stress-dependent characteristics show soft magnetic behavior at low stress. But the wire becomes magnetically harder at higher stress values. Similar behaviors have also been observed for samples annealed at different stresses. For annealed samples the softness extends to higher stress-annealed values than that observed from the stress behavior of the as-quenched state. The anisotropy induced during flash annealing under tensile stress is responsible for the change in magnetic properties. So  $H^*$  and  $M^*$  in the as-quenched state can be modified by choosing the appropriate value of stress applied during flash annealing. This characteristic can be used for pulse-generating sensor elements.

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