

SC-R-67-1224
February 1968

Reprint

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OF INVAR AND SILECTRON FROM 30-450 KBAR

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Pressure Dependence of the Magnetization of Invar and Silectron from 30-450 kbar*

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Measurements of the change in saturation magnetization of Invar and Silectron subjected to shock-wave compression from 30 to 450 kbar show that Invar exhibits a constant coefficient of saturation magnetization change with pressure, $M_s^{-1}(dM_s/dp)$ and that Silectron experiences a pressure-induced transition to a nonferromagnetic phase. Shock waves are generated in tape-wound core samples by projectile impact techniques which allow experiments in small pressure increments over a wide range in pressure. The Invar measurements give a value for $M_s^{-1}(dM_s/dp)$ of -1.3×10^{-2} kbar $^{-1}$ up to a magnetization change of 90% of the saturation magnetization. This value is the same as that obtained in previous static measurements to 5 kbar. Measurements on Silectron (grain-oriented 3% Si-97% Fe) cores show a change in magnetization beginning at 150 kbar which is the pressure at which a first-order transition has been detected from previous shock-wave pressure-volume measurements. The present measurements indicate that the high-pressure phase of Silectron is nonferromagnetic and show that a mixed-phase region extends to a pressure of 225 kbar. These shock-wave measurements cover a pressure range which is about two orders of magnitude greater than that used previously in static magnetization vs pressure measurements.

INTRODUCTION

Recently, we reported shock-wave compressibility measurements that showed a pressure-induced Curie-point transition in fcc 30% Ni-70% Fe at a pressure of 22.6 kbar.¹ Since shock-wave experiments achieve much higher pressures than are currently possible with static techniques, the agreement between these results and previous lower pressure hydrostatic values² encouraged high-pressure magnetization measurements. In the present paper saturation magnetization measurements are reported for Invar (36% Ni-64% Fe)

and Silectron (grain oriented 3% Si-97% Fe) subjected to shock-wave pressures in the range of 30 to 450 kbar. Previous experiments with Silectron³ and a nickel ferrite⁴ have demonstrated that appreciable magnetization changes result from high-pressure shock waves.

EXPERIMENTAL

When a toroidal ferromagnetic sample is subjected to shock loading a shock wave with pressure, p , moves through the sample with a velocity, U . An N -turn coil with inductance, L , is wound around the sample and connected to a resistive circuit such that the L/R time constant is long compared to the time required for the shock wave to traverse the sample thickness, l . In

* This work was supported by the United States Atomic Energy Commission.

¹ R. A. Graham, D. H. Anderson and J. R. Holland, *J. Appl. Phys.* **38**, 223 (1967).

² J. S. Kouvel, in *Solids Under Pressure*, W. Paul and D. M. Warschauer, Eds. (McGraw-Hill Book Company, Inc., New York, 1963).

³ R. W. Kulterman, F. W. Neilson, and W. B. Benedick, *J. Appl. Phys.* **29**, 500 (1958).

⁴ E. B. Royce, *J. Appl. Phys.* **37**, 4066 (1966).

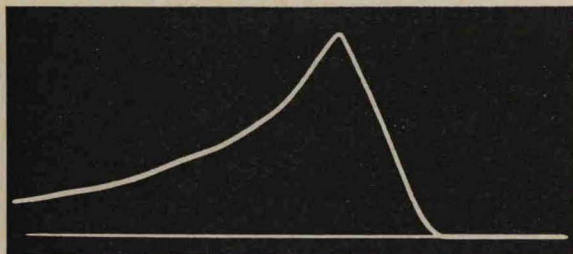


FIG. 1. Current-time record for Silectron impacted at 425 kbar. Time increases from right to left. The peak current (765 A) is proportional to the magnetization change. The time from first signal to peak current is the shock-wave transit time of 1.06 μ sec.

this configuration the current, i , in the coil is:

$$i(t) = w(N/L)(\Delta M U)t \quad 0 < t < l/U \dots \quad (1)$$

where t is the time, ΔM is the change in magnetization and w is the sample width. Thus, the current is proportional to U and ΔM and when $t=l/U$ the current is a direct measure of ΔM . The typical current-time waveform shown in Fig. 1 shows that the response is, to a close approximation, that predicted by Eq. (1).

The currents resulting from the various shock-wave pressures were measured in separate experiments on samples which were saturated with an exciting coil and whose dimensions and coil configurations were nominally the same. Thus problems in defining absolute values for N and L were avoided and the peak current (at $t=l/U$) served as a relative measure of the saturation magnetization change at each pressure. Upon complete demagnetization the peak current no longer increased with increases in pressure. This maximum current value corresponds to the saturation magnetization change, M_s , to which the other current values were normalized.

Eddy-currents are minimized by the use of commercially available 0.0016-cm-thick insulated tape, wound into a high-density core of rectangular cross section. The impact occurs on an 18 \times 18 mm flat surface of the core and the resulting shock wave propagates through one of the 5-mm-thick legs of the core. The pressure and pressure-time history depend directly upon the mechanical response of the sample and, as will be determined later, the response of this laminated configuration is suitable for shock-wave experiments.

Different pressure amplitudes were produced by the controlled impact (in vacuum) of projectiles on the samples. The pressure imparted to the sample depends upon the velocity (which is measured to $\pm \frac{1}{2}\%$) as well as the mechanical properties of the projectile and sample.⁵

⁵ The pressures are known to $\pm 6\%$ in absolute amplitude and $\pm 3\%$ in relative amplitude. For a more complete description of impact experiments see: R. A. Graham, ASME Preprint 66-WA/PT-2, Nov. 1966.

INVAR

Results of current measurements on the Invar samples⁶ at pressures from 30 to 210 kbar are shown in Fig. 2. A value of $M_s^{-1}(dM_s/dp)$ of $1.3 \pm 0.1 \times 10^{-2}$ kbar⁻¹ is obtained over the major portion of the pressure range.⁷ This value is in excellent agreement with results of low-pressure hydrostatic measurements² and hydrostatic measurements to 25 kbar on specimens in this tape-wound configuration.⁸ For pressures close to that required for complete demagnetization the observed response is similar to that observed in magnetization vs temperature measurements near the Curie temperature.

The results show that the coefficient of saturation magnetization change with pressure is constant with pressure and that the tape-wound core construction can be used for shock-wave experiments conducted above 30 kbar.

SILECTRON

Similar measurements on Silectron samples^{9,10} at pressures from 50 to 450 kbar show a much different behavior. Small current values consistent with the low coefficient of magnetization change with pressure are observed at 50 and 120 kbar. However, at a pressure of 150 kbar larger current values are observed. These currents increase in magnitude until a pressure of 375 kbar is reached. The pressures at which significant changes in response occur indicate that the large magnetization changes are the result of a previously detected pressure-induced first-order phase transition.⁹

The volume change resulting from this transition causes two separate shock waves to propagate from a given pressure input to the sample. The reflection of the leading wave from the rear of the sample prevents the entire thickness of the sample from experiencing the high pressure behind the second wave. At a pressure of 375 kbar, however, the velocity of the second

⁶ The Invar samples were impacted by aluminum projectiles. The mechanical properties of aluminum were taken from: J. M. Walsh, M. H. Rice, R. G. McQueen and F. L. Yarger, Phys. Rev. **108**, 196 (1957); G. R. Fowles, J. Appl. Phys. **32**, 1475 (1961); and C. D. Lundergan and W. Herrmann, J. Appl. Phys. **34**, 2046 (1963). The mechanical properties of Invar were taken from: D. R. Curran, J. Appl. Phys. **32**, 1811 (1961).

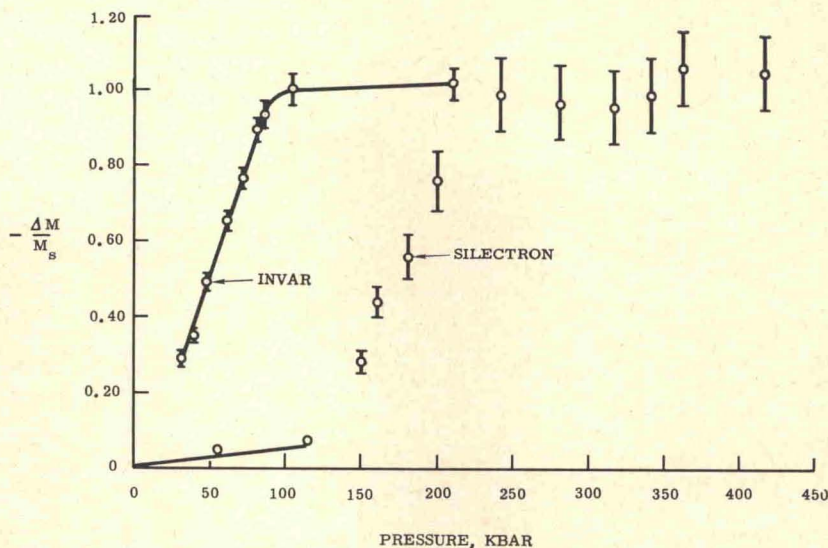
⁷ The temperature change induced by the shock wave ($< 10^\circ\text{C}$ at 100 kbar) has a negligible effect.

⁸ G. A. Samara (private communication).

⁹ The Silectron samples were impacted by 34% Si-Fe projectiles for experiments up to 280 kbar. This composition is nominally the same as Silectron. The mechanical properties of this alloy were taken from: E. G. Zukas, C. M. Fowler, F. S. Minshall, and J. O'Rourke, Trans. AIME **227**, 746 (1963). For experiments above 300 kbar Mallory 1000 projectiles ($\rho_0 = 16.83 \text{ g/cm}^3$) were used. The mechanical properties were assumed to be that observed for tungsten when adjusted for the 12% difference in density. Tungsten properties were taken from R. G. McQueen and S. P. Marsh, J. Appl. Phys. **31**, 1253 (1960).

¹⁰ The temperature change induced by the shock wave ($< 40^\circ\text{C}$ at 150 kbar) has a negligible effect.

FIG. 2. Relative magnetization change of Invar and Silectron at various pressures. The data on Invar indicate that below 30 kbar the response is influenced by the elastic limit of the material and the laminated construction of the sample.



wave becomes equal to that of the first, a single shock wave is formed, and the entire sample is demagnetized. Thus, the peak currents observed between 150 and 375 kbar are influenced by various nonuniform pressure distributions in the *samples* as well as various magnetization changes of the *material*. In this case, the peak currents predicted from Eq. (1) are incorrect. With some reduced accuracy, however, we can measure values of di/dt from the current-time waveforms for times before the wave reflection occurs. These values are proportional to the product of the magnetization change and the velocity of the higher-pressure wave. The magnetization change shown in Fig. 2 is computed from these di/dt values and the wave velocity as obtained from the previous shock-compression experiments.⁹ Again, the higher-pressure di/dt values are used as the reference to which all the lower-pressure points are normalized.

These data show that when a pressure of 225 kbar

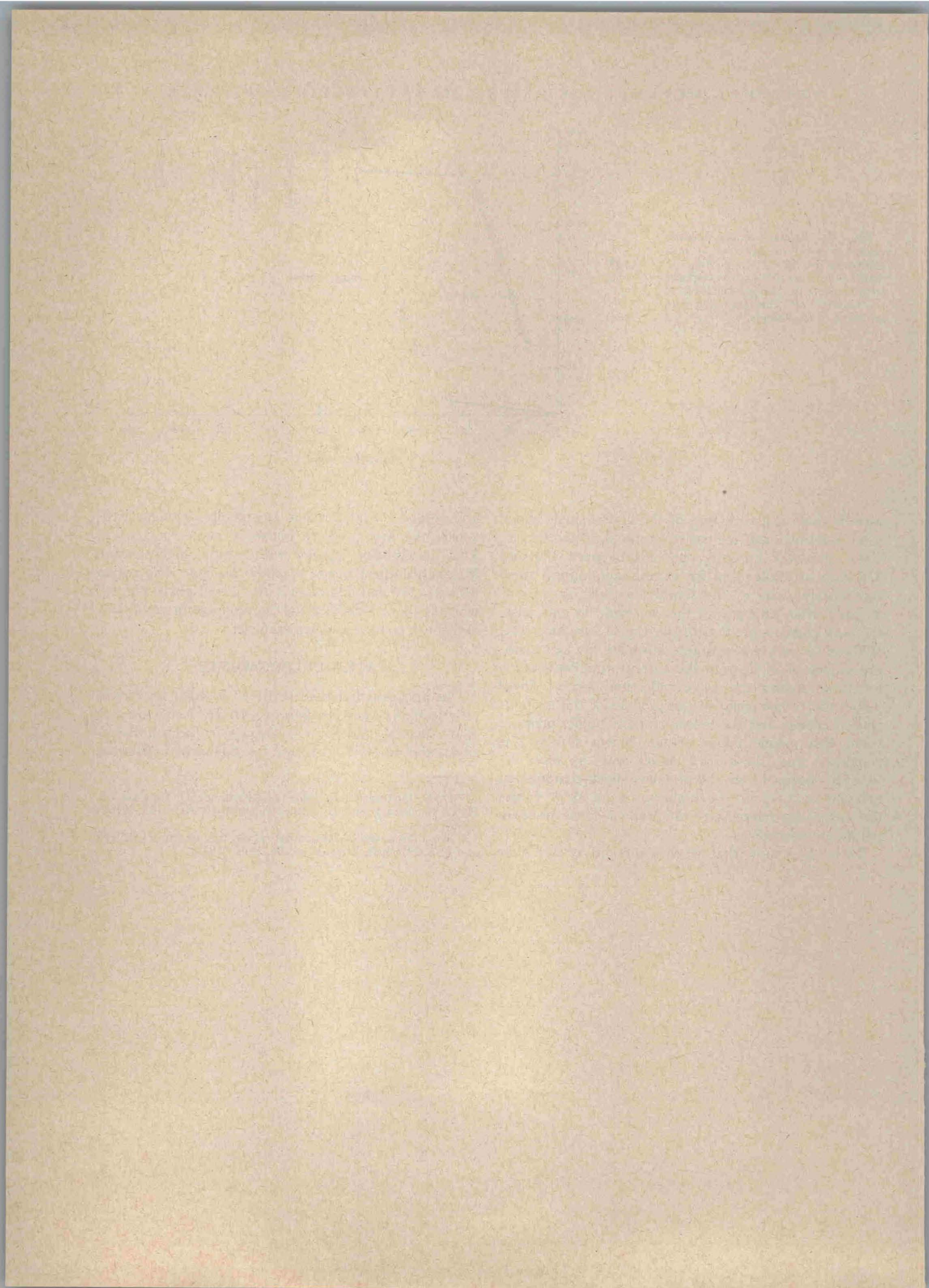
is exceeded the material is completely demagnetized; hence, the high-pressure phase is nonferromagnetic. This observation agrees with static high-pressure Mössbauer effect measurements¹¹ on iron. The region between 150 and 225 kbar is the mixed-phase region¹² which is characteristic of the thermodynamic conditions of a shock-compression experiment.

ACKNOWLEDGMENTS

The author is indebted to B. A. Worden for especially valuable contributions, to D. H. Anderson and R. C. Wayne, and other colleagues for helpful discussions, and to G. E. Ingram for technical assistance.

¹¹ D. N. Pipkorn, C. K. Edge, P. Debrunner, G. De Pasquali, H. G. Drickamer, and H. Frauenfelder, *Phys. Rev.* **135**, A1604 (1964).

¹² For a discussion of these mixed-phase regions see: R. E. Duff and F. S. Minshall, *Phys. Rev.* **108**, 1207 (1957).



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