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High-Pressure Phase Transition and Demagnetization in Shock Compressed Fe-Mn Alloys*

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Shock deformation of Fe-Mn alloys up to 14 wt% Mn results in a shock-induced phase transformation. It has been shown that bcc martensite with manganese in the range 4–16 wt% transforms to a stable close-packed phase under pressure without the occurrence of reversion. The addition of manganese to iron also decreases the transition pressure from 133 kbar for pure Fe to less than 70 kbar for Fe–14Mn. X-ray diffraction, electron-probe microanalysis, electron microscopy, and density results indicate that for the Fe-4Mn and Fe-7Mn alloys, the fcc phase has been stabilized after shock deformation, while the ϵ phase has been stabilized for the Fe-14Mn alloy. Saturation magnetization studies have detected a residual reduction in magnetization due to the retainment of the high-pressure phase.

I. INTRODUCTION

A magnetic phase transition in iron in the vicinity of 130 kbar has been confirmed in both dynamic and static experiments.[1–3] Recent experiments indicate that a partial phase transition to the nonmagnetic ϵ phase (hcp) can occur at shock pressures as low as 50 kbar.[4] However, since iron reverts to its initial structure on relief, residual phase changes have not been observed.

The temperature-pressure diagram for iron, shown in Fig. 1, can be modified by the addition of alloying elements to iron. Specifically, the stability of the close-packed phase of iron can be increased by the addition of manganese.[5] The phase diagram is shown in Fig. 2. Magnetic susceptibility measurements[6] of Fe-Mn alloys that had been shocked at pressures up to 300 kbar indicated that the high-pressure phase could be retained at zero pressure. Since the duration of a shock-wave pressure pulse is about 10^{-6} sec, any phase transformation which occurred must be martensitic.

The present work is an investigation of the residual magnetic and crystallographic phase changes in shock-deformed iron-manganese alloys. Our experimental results are related to shock-induced martensitic and magnetic transformations. We will show that in Fe-Mn density, x-ray microstructural, and magnetization changes are due to the partial retention of the high-pressure phase.

II. EXPERIMENTAL DETAILS

A. Sample Preparation

The composition of the alloys included in this study is given in Table I. All alloys were prepared using an electrolytic grade of iron and high-purity manganese (99.9%); 0.007 wt% carbon was the main im-

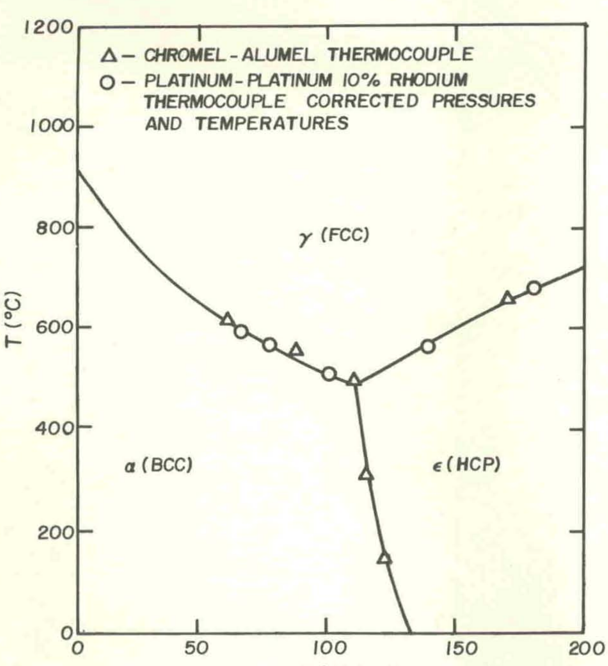

FIG. 1. Temperature-pressure diagram for iron (see Ref. 2).

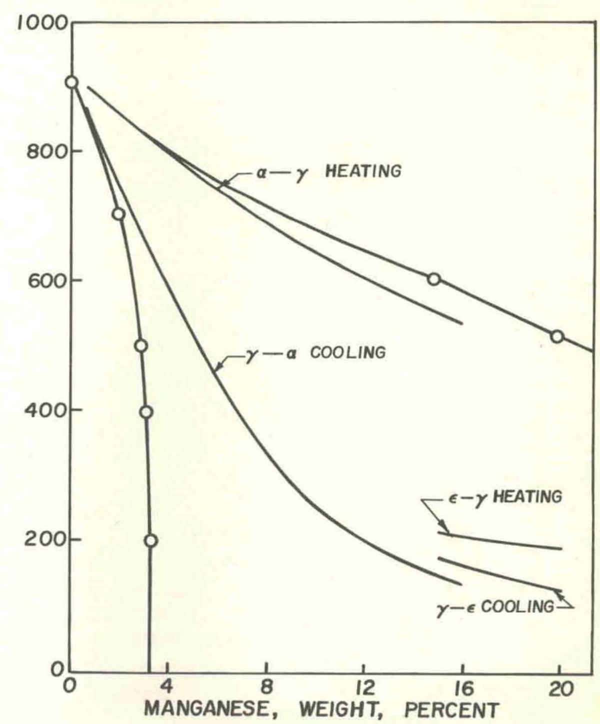

FIG. 2. Transformation and equilibrium diagram for the Fe–Mn system.

TABLE I. Composition of alloys.

Alloy designation	Composition (wt% Mn)
Fe	0
Fe-0.4Mn	0.38
Fe-4Mn	4.10
Fe-7Mn	7.37
Fe-14Mn	13.62

purity. The alloys were arc melted in an argon atmosphere under conditions which insured good homogeneity. Hot-rolled samples having dimensions $20\times6.0\times1.3$ mm were machined and annealed for 9 h at temperatures between 900–950 °C. Half of the specimens were quenched from the austenitic region and the rest were furnace cooled to room temperature in 72 h. Plane-wave shock-deformation tests were performed at peak pressures of 90, 150, 300, and 500 kbar. The basic method of plane-wave generation has been described previously.[7]

B. Experimental Methods

Magnetization curves for shock deformed and annealed specimens were determined by standard "ring techniques" described by Rose, Villere, and Berger.[8] All measurements were taken at room temperature. The maximum applied field was 1000 Oe.

Density was measured at the National Bureau of Standards and at the Naval Weapons Laboratory using a displacement technique. Density changes of ±0.0001 g/cm^3 were detectable. The liquid used in determining the density was Di(2-ethyl hexyl) azelate.

Crystal structures and microstructures were observed with x-rays, electron probe, light, and transmission electron microscopy.

The electron probe was used to study variations of manganese concentrations and to facilitate the interpretation of the optical micrographs. The Norelco instrument was operated at 30 kV with a specimen current between 0.035 and 0.05 μA. To determine the manganese content within the bcc and close-packed phases, a calibration curve of relative intensity $(I/I_0)_{\mathrm{Mn}}$ vs manganese content was determined using four homogeneous Fe-Mn alloys (Fig. 3). It was of interest to show that the calibration curves agree with the calculated curves of Castaing[9] and Wittry[10] who made corrections for absorption and fluorescence.

Identification of the retained high-pressure was accomplished by x-ray diffraction of powdered Fe-Mn using Mo$K\alpha$ radiation. Thin foils, suitable for examination by electron transmission microscopy, were electropolished in a solution consisting of 430 ml of H_3PO_4, 50 g of CrO_3, and 7 ml of distilled water at 0 °C and a current density of 1.2 A cm^{-2}.

III. EXPERIMENTAL RESULTS

A. Density Experiments

Density measurements are shown in Table II. Density measurements of the quenched and shock-loaded

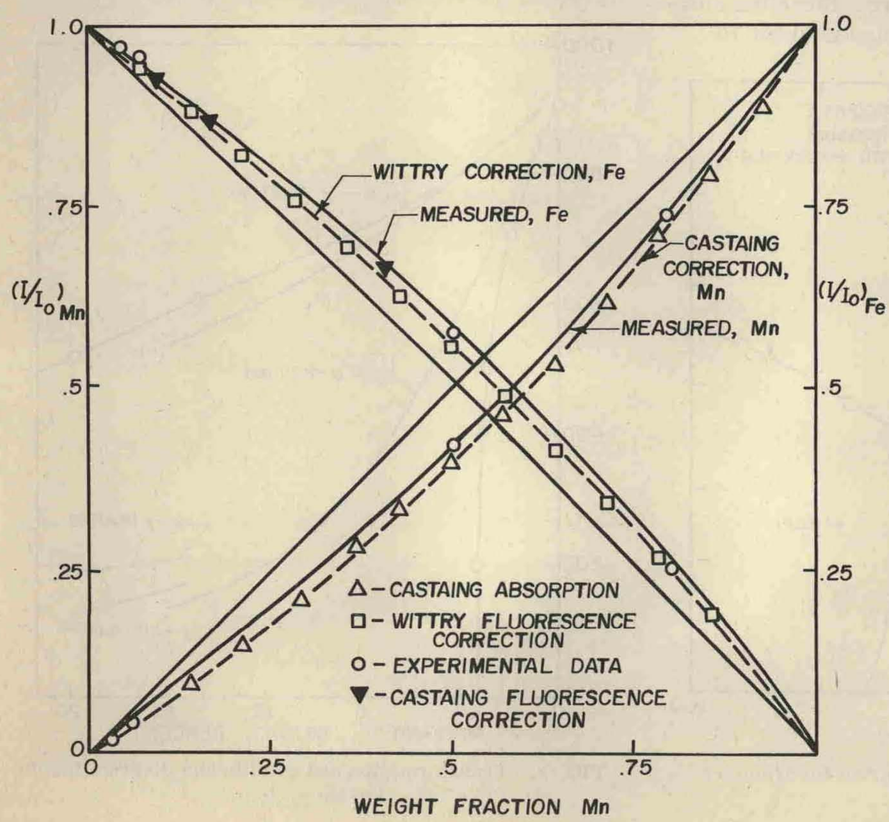

FIG. 3. Measured I/I_0 vs composition curves for the Fe-Mn system.