Dynamic properties and microstructural behaviour of Armco iron shock-loaded at high temperatures
H. Nahme, M. Hiltl
Fraunhofer-Institut für Kurzzeitdynamik, Ernst-Mach-Institut, Eckerstrasse 4, D-79104 Freiburg, Germany

Abstract

Using planar-plate impact technique in combination with a velocity interferometer VISAR dynamic properties of Armco iron at sample temperatures of $290 \, \text{K} < T < 1050 \, \text{K}$ have been determined. At temperatures above $800 \, \text{K}$ the $\alpha$-$\gamma$ phase transition has been observed, when pressure stresses exceeding $9 \, \text{GPa}$ were applied. Softly recovered samples have been examined by means of optical and SEM microscopy. At temperatures and stress levels sufficient for the $\alpha$-$\gamma$, $\alpha$-$\varepsilon$ and $\gamma$-$\varepsilon$-phase transition, the samples show considerable changes in the structural condition.

The samples used for the planar-plate-impact tests may be regarded as very simple structure elements. The experimental results will show, that although the structure is very simple, surprising results may arise from the alternating effects of shape, size and material properties.

1 Introduction

Since the pressure induced phase transition $\alpha \rightarrow \varepsilon$ in pure iron was reported by Bancroft et al.\textsuperscript{1} the phase transitions $\alpha$ (bcc) $\rightarrow$ $\varepsilon$ (hcp) [Barker & Hollenbach\textsuperscript{2}, Bundy\textsuperscript{4}, Arnold\textsuperscript{6}], $\alpha$ (bcc) $\rightarrow$ $\gamma$ (fcc) [Johnson et al.\textsuperscript{3}, Bundy\textsuperscript{4}] and $\gamma$ (fcc) $\rightarrow$ $\varepsilon$ (hcp) [Bundy\textsuperscript{4}] have been described by several authors. Most of these investigations concentrated on the $\alpha$-$\varepsilon$-phase transition and have been performed by means of quasistatic tests [Bundy\textsuperscript{4}], plate impact [Barker & Hollenbach\textsuperscript{2}, Arnold\textsuperscript{6}] and laser shock [Romain et al.\textsuperscript{5}] Only few dynamic tests have been done at high temperatures [Johnson et al.\textsuperscript{3}].

In the report given here the dynamic properties of Armco iron shocked at different temperatures and the microstructural response to the loading process
are described. Dynamic material data determined at room temperature taken from the work of Arnold are used as a standard for comparison purposes.

2 Material description

All samples have been made from an Armco iron rod. The material has been investigated as received, no heat treatment or cold working has been done prior to the loading process. The grain size of the material was 80-100 μm, the density was 7850 kg/m³. Longitudinal and transvers sound velocity have been determined to be 5950 m/s and 3400 m/s respectively.

The samples were circular plane parallel plates of 50 mm diameter and 8mm thickness for all tests described here. Projectile plates of 58mm diameter and 3-4mm thickness made of the same material have been used.

3 Experimental setup

The planar plate impact technique has been used in combination with a VISAR [Barker & Hollenbach]. The experimental setup is shown schematically in fig 1. The projectile plates have been accelerated by means of a single stage gas gun.

For high temperature tests an expendable furnace unit was used. The sample temperature has been measured with an accuracy of ±15K using NiCr-Ni thermocouples at the rear side of the sample.

For temperatures above 570K the samples were coated by a thin (1-2.5 μm) ceramic layer to avoid corrosion. Very little degradation of the reflecting surface quality has been observed with these coatings.

To provide free interaction of all waves created by the impact process, no windows or backing and no lateral momentum trap has been used and care has been taken to have a free rear surface at the central part of the projectile.

All samples have been recovered softly after shock loading for examination of the microstructure by means of optical and SEM microscopy.
4 Data reduction

In the following the term $P_2^*$ is used for the plastic wave propagating in $\gamma$ - iron in analogy to the $P_2$ - wave of $\varepsilon$ - iron used by Barker & Hollenbach.

From the VISAR data the Hugoniot elastic limit $\sigma_{HEL}$, shock velocity $U_S$, shock velocities $U_{P2}$, $U_{P2^*}$ (shock velocity in the $\varepsilon$ and $\gamma$ - phases respectively), spall strength $\sigma_{sp}$, phase transition stresses $\sigma_{a\rightarrow\varepsilon}$ and $\sigma_{a\rightarrow\gamma}$ and the maximum stresses $\sigma_{max}$ have been determined using the following equations:

$$\sigma_{HEL} = \frac{1}{2} \rho(T) c_1(T) \Delta u_{HEL}$$  \hspace{1cm} (1)

$$U_S = c_1(T) / (1 + \left( \frac{c_1(T) \Delta t}{d_T} \right))$$  \hspace{1cm} (2)

$$U_{P2} = U_S \left( \frac{2d_T - U_S \Delta t_{P2}}{2d_T + U_S \Delta t_{P2}} \right)$$  \hspace{1cm} (3)

$$U_{P2^*} = U_S \left( \frac{2d_T - U_S \Delta t_{P2^*}}{2d_T + U_S \Delta t_{P2^*}} \right)$$  \hspace{1cm} (4)

$$\sigma_{sp} = \frac{1}{2} \rho(T) U_S \Delta u_{sp}$$  \hspace{1cm} (5)

$$\sigma_{a\rightarrow\varepsilon} = \sigma_{HEL} + \rho(T) U_S \left( \frac{\left( u_{a\rightarrow\varepsilon} - \Delta u_{HEL} \right)}{2} \right)$$  \hspace{1cm} (6)

$$\sigma_{a\rightarrow\gamma} = \sigma_{HEL} + \rho(T) U_S \left( \frac{\left( u_{a\rightarrow\gamma} - \Delta u_{HEL} \right)}{2} \right)$$  \hspace{1cm} (6a)

$$\sigma_{max} = \sigma_{HEL} + \sigma_{a\rightarrow\varepsilon} + \rho(T) U_{P2} \left( \frac{\left( u_{max} - u_{a\rightarrow\varepsilon} \right)}{2} \right)$$  \hspace{1cm} (7)

$$\sigma_{max} = \sigma_{HEL} + \sigma_{a\rightarrow\gamma} + \rho(T) U_{P2^*} \left( \frac{\left( u_{max} - u_{a\rightarrow\gamma} \right)}{2} \right)$$  \hspace{1cm} (7a)

with the following notations:

- material density $\rho(T)$, (temperature dependent)
- longitudinal sound velocity $c_1(T)$, (temperature dependent)
- amplitude of the elastic precursor $\Delta u_{HEL}$
- amplitude of the phase transition signals $u_{a\rightarrow\varepsilon}$, $u_{a\rightarrow\gamma}$
- time interval between velocity increase due to the elastic and plastic waves $\Delta t$
- target thickness $d_T$
- time intervals between velocity increase due to the plastic wave and the respective $P_2$ and $P_2^*$ - waves in $\varepsilon$ and $\gamma$ - phase iron $\Delta t_{P2}$ and $\Delta t_{P2^*}$
- amplitude of the spall signal $\Delta u_{sp}$.

The high temperature values of the density and longitudinal sound velocity necessary for these calculations have been taken from work of Pepperhoff.

For the calculation of the spall strength the shock velocity $U_S$ has been used, because due to the small $\sigma_{HEL}$ the plastic wave component provides the main contribution to the spall process.

To determine the contribution of the $P_2$ and $P_2^*$ waves to the maximum stresses, the density of the iron in the $\gamma$ and $\varepsilon$ - phases have been assumed to be

$$\rho_{\gamma}(T) = \rho_{\varepsilon}(T) \frac{n_{\varepsilon,hcp}}{n_{\varepsilon}}$$  \hspace{1cm} (8)

with the closest package density $n_i$ of the three crystal phases.
5 Experimental results and discussion

5.1 Dynamic properties

In fig. 2 the pressure - temperature diagram of pure iron adopted from the work of Giles et al. is displayed together with the pressure stress data of all tests. For those tests yielding a sufficient VISAR signal to determine the phase transition stresses, the phase transformation data are shown as solid dots, the final p-T-states of these tests are marked by crosses. For tests without VISAR-data the final stress - temperature states expected from the impact conditions are given by open squares. The knowledge of the final state is necessary for the interpretation of the microstructure described in section 5.

Figure 2: p-T diagram of pure iron with pressure stress states of this work

From these data the tests may be divided into four groups:
1. The loading path includes no phase transition
2. The loading path includes the $\alpha \rightarrow \varepsilon$ - phase transition
3. The loading path includes the $\alpha \rightarrow \gamma$ - phase transition
4. The loading path includes the $\alpha \rightarrow \gamma$ and the $\gamma \rightarrow \varepsilon$ - phase transitions

In fig. 3 velocity time histories of some tests at high temperatures are displayed. All velocity curves show the typical time course with the elastic precursor followed by the plastic wavefront. From the amplitude of the elastic precursor a Hugoniot elastic limit of $0.75 \text{ GPa} < \sigma_{\text{HEL}} < 1.0 \text{ GPa}$ is determined for temperatures around 830K with a tendency of lower values at higher temperatures. The considerable reduction of the elastic limit of 40% with respect to the corresponding value of Armco 80 at room temperature of 1.3 GPa [Arnold] cannot be explained by the temperature induced decrease of density and sound speed only. Also the amplitude of the elastic precursor is reduced at high temperature. This may be due to a more pronounced precursor decay at high temperatures but has to be confirmed by further experiments.
The shock velocity $U_S$ is also compared to the values known for Armco 80 in fig. 4. The shock velocity is reduced for about 12% only, which is very similar to the decrease of the longitudinal sound speed with increasing temperature.

The steep velocity increase due to the plastic wave is followed by a plateau indicating either the $\alpha \rightarrow \gamma$ or $\alpha \rightarrow \varepsilon$ phase transition. From the p-T diagram it can be seen, that the stress required for the $\alpha \rightarrow \gamma$ transition strongly depends on the sample temperature. The stress values determined for this transition are in reasonable agreement with the phase diagram adopted from the work of Giles. From the velocity increase following the plateau the propagation velocity of the $P_2^*$ and $P_2$-waves were determined using the eq. 3 and 4 respectively. The values are plotted in fig. 4 together with the data for Armco 80 for the $\alpha \rightarrow \varepsilon$ transition [Arnold].

The spall strength has been determined using eq. (5). From the Lagrange diagram (fig.7) it is expected that the main spallation occurs while the iron is in the $\varepsilon$ or $\gamma$-phase. Because the bulk sound speed is not known for these phases in a first attempt $U_S$ was used for the calculation, yielding spall strengths in the range $1.5 \text{GPa} < \sigma_{sp} < 2.5 \text{GPa}$ for $\gamma$-iron and $1.7 \text{GPa} < \sigma_{sp} < 2.3 \text{GPa}$ for $\alpha$-iron around 830K. This leads to the conclusion that the material is of comparable spall strength in all three phases. Differences in the temporal evolution of the spall signals for samples being stressed to below and beyond the phase transformation limit respectively can not be explained yet.

In the case of the loading path $\alpha \rightarrow \gamma \rightarrow \varepsilon$ at certain impact conditions the three wave structure inside the sample has to be replaced by a four wave structure with an even more complex influence to the microstructural behaviour. For example the number of spall planes may be increased further (see below).
From the shock velocities measured for these two phases it seems possible, that at least at certain conditions a further step in the velocity - time - history may be visible. In some of these tests there is an indication, that also the \( \gamma \rightarrow \varepsilon \) transition shows up in the VISAR-signal but the signals are heavily distorted at the exact time, when this phase transition is expected to occur. This distortion of the signal is mostly caused by the formation of a central hole in the samples which is described in more detail in section 5.2.

5.2 Microscopic examination

All samples have been recovered softly for microscopic examination. The following description of the microstructural behavior is divided into 4 sections according to the classification of the tests given in section 5.1.

5.2.1 Loading path includes no phase transition  Samples not being affected by a phase transition showed deformation twins typical for shock loaded iron. At high temperatures the twin density was strongly reduced. This is attributed to the competitive processes of twinning and dislocation with the latter being favoured at high temperatures and is in good agreement with theoretical predictions of Meyers & Murr\textsuperscript{10}.

Because no window or backing technique was used all samples showed spallation.

Samples being shocked at temperatures above 560 K showed the formation of voids and void-like structures (fig 5a,b). A very pronounced concentration of these voids, finally forming the spall plane indicates, that these voids were produced while the material was subjected to tensile stress.

The production process of the voids is not yet clear. Very often there are small areas of material, separated completely from the surrounding material. These areas often show the structure of the surrounding material with respect to twinning and slip lines, indicating, that the voids are created at a later stage of the deformation process.
5.2.2 Loading path including the $\alpha \rightarrow \varepsilon$ phase transition  
Samples shock loaded at room temperature to stresses above the $\alpha$-$\varepsilon$-phase transition stress level showed the well-known change of the microstructure (fig. 6a), indicating stress levels sufficient for the phase transition to occur [Romain et al.]. For samples being transformed to the $\varepsilon$-phase the location of the spall plane shifted from the expected position and in some cases additional spall planes have been found. The additional spall planes can be explained as a result of the three wave structure in iron, shocked to stresses above 13 GPa as can be seen from fig. 7. Depending on impact conditions and propagation velocities of the different waves, these additional spall planes occur at different locations. The observed shift of the main spall plane towards the impacted side of the sample can not be explained at present.

Some samples impacted at velocities around 1000 m/s at room temperature showed two regions of completely different microstructure at the spall plane. A central smooth area of ductile fracture behavior (fig. 6b) was surrounded by an area showing brittle fracture (fig. 6c). In iron the fracture mechanism is known to depend on sample temperature and strain rate. While increasing temperature causes ductile behavior, increasing strain rate causes brittle fracture behavior. Because the strain rate should be the same across the sample, the distribution of the ductile and brittle fractured areas indicates an increased temperature in the center of the sample. This is further confirmed by the observation of a hole in the samples center at high impact velocities and by the microstructure found in the central areas of the samples.
5.2.3 **Loading path including the \( \alpha \rightarrow \gamma \) - phase transition** In samples shocked to p-T-states within the \( \gamma \) - phase region areas of very fine grained structure appeared. These fine grains are attributed to a dynamic recrystallization process [Humphreys & Hatherly\(^{11}\)] , that is favoured by increasing dislocation mobility at high temperatures. These areas become larger with increasing temperature and impact velocity until the whole sample is recrystallized. At temperatures and pressures just sufficient to achieve the \( \alpha \rightarrow \gamma \) - transition the recrystallization occurs primarily near the spall plane. Here voids and microcracks are surrounded by fine grained areas and are connected by chains of small grains of 5-10 \( \mu \text{m} \) diameter. This and the fact, that the recrystallization in other samples seem to have started near the spall plane indicates, that tension stress prior to the spallation initiates recrystallization. Indeed, in several samples instead of additional spall planes chains of small crystals were found at locations comparable to additional spall planes in other samples (fig. 8).

As in the case of the \( \alpha \rightarrow \varepsilon \) transition the spall plane shifted and additional spall planes occured at higher stresses. Again additional planes may be explained by the triple wave structure (fig. 7). Because the spall plane which occurs nearest to the sample rear surface will prevent all other spallation processes from being observed by VISAR measurements, the temporal evolution of the different planes is not yet clear. Nevertheless the investigation of the microstructure clearly shows, that additional spall planes are created.
5.2.4 Loading path including the $\alpha \rightarrow \gamma$ and the $\gamma \rightarrow \varepsilon$ - phase transitions

The most complex microstructure was observed when both the $\alpha$-$\gamma$- and the $\gamma$-$\varepsilon$-phase transitions occurred. All microstructural features described so far have been found in a single sample.

- The main part of the samples revealed the needle-like structure when stress and temperature were not too high.
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• With increasing temperature and stress the amount of recrystallized areas grew until the needle-like structure is totally converted to the fine grained structure by recrystallization at temperatures above 970K and/or stresses above approximately 18 GPa.

• Near the rear surface the stress was not sufficient for the $\gamma - \varepsilon$- or even the $\alpha - \gamma$-phase transition. So in these areas the grains only showed the very low twin density known from the high temperature tests described before.

• The voids in these samples were located mainly near the main spall plane and at a lower density in areas where additional spall planes occurred.

It seems that even where the amplitudes of the release waves alone are not sufficient, the concerted acting of release waves and phase transitions may cause spallation.

A very surprising feature was the formation of a central hole through one or both parts of the sample plate and in some cases also through the projectile plate. The origin of the central hole is not yet fully understood. In all but one experiments the VISAR-signal was heavily disturbed and no statement from the point of dynamic measurements was possible about the processes finally leading to the formation of this hole. The process responsible for the holes obviously started near the spall plane and was directed outward. First the spalled part of the sample is affected, but the processes involved finally led to penetration of all plates, when stress and temperature were high enough. In a single test with 800m/s impact velocity at room temperature the VISAR-signal contained information about a very strong acceleration of the material at the sample center at the exact time, when the $P_{2\gamma}$-wave was expected to show up at the sample rear surface.

In all cases when a central hole was found, the microstructure of the recovered sample indicated, that material was ejected in a very violent process. Recrystallization near the edges of this hole and the differences in the fracture mechanism leading to the spall surface indicates, that the phase transition $\alpha \rightarrow \varepsilon$ and $\gamma \rightarrow \varepsilon$ provide an important contribution to the processes involved.

6 REFERENCES


5. Romain, J.P. Hallouin, M. Gerland, M. Cottet, F. Marty, L. $\alpha \leftrightarrow \varepsilon$ Phase Transition in Iron Induced by Laser Generated Shock Waves, in Shock


