Original Article

Microstructures and properties of solid and reticulated mesh components of pure iron fabricated by electron beam melting

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This research examines rapidly solidified, atomized pure iron powder and solid and reticulated mesh components fabricated by electron beam melting (EBM) from this powder precursor. Especially significant was the characterization of associated microstructures and corresponding mechanical properties. Atomized Fe powder was used to fabricate solid and reticulated mesh components by EBM. Powder and component microstructures and phase structures were examined by light (optical) metallography, scanning electron microscopy, X-ray diffractometry, and transmission electron microscopy. Corresponding Vickers microindentation hardness measurements were also made and compared to tensile data along with measurements of dynamic stiffness for mesh components having varying densities. The atomized Fe powder was observed to contain δ-Fe which was retained in the solid, EBM-fabricated components where it was observed to be homogeneously distributed in equiaxed α-Fe grains as δ-phase platelets measuring ~0.5 μm to 2 μm in length and ~40 nm thick; coincident with the α-Fe matrix {100} or {110} planes. A log-log plot of E/E0 versus ρ/ρ0 resulted in (E/E0) = (ρ/ρ0)α. Novel, δ-Fe phase platelets have been observed in α-Fe components fabricated by EBM.

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1. Introduction

Iron represents one of the most abundant and historically oldest metal used by humans, beginning roughly 3500 years ago at the start of what is often called the Iron Age. Basic iron production world-wide generally exceeds 1 billion tons [1]. Iron and steel account for roughly 80 percent of all metal powders produced annually, and iron powder represents the largest tonnage of raw materials used in powder metallurgy (P/M) fabrication [2].

The mechanical properties of iron are sensitive to purity and tensile and yield strengths can vary from ~150 to 300 MPa and 90 to 150 MPa, respectively. Young’s modulus has been recorded from less than 100 GPa to 200 GPa. Tensile elongations have been shown to vary from 15 to 25 percent.

Iron has three allotropic phases: δ-phase (bcc) between the melting point at 1537 °C to ~1390 °C, γ-phase (fcc) between ~1390 °C to 910 °C, and α-phase (bcc) below 910 °C. The lattice constant for δ-Fe and α-Fe varies linearly from ~2.93 Å to 2.87 Å, and from 2.87 Å to 2.82 Å, respectively, interrupted by the γ-Fe phase between 1390 °C and 910 °C, where the lattice parameter varies from 3.68 Å to 3.63 Å [3].

As a high-temperature phase, δ-Fe has no influence on iron metallurgy, and its microstructure has not been well documented. α-Fe is usually characterized by irregular, equiaxed grain structure which can be better controlled in P/M processing which is employed to produce high wear iron products with small grain sizes [2]. At the Curie point (770 °C) α-Fe becomes magnetic.

In this study, the fabrication of iron components from atomized, rapidly solidified, high-purity iron precursor powder was explored using electron beam melting (EBM). Special interest involved the observations of the precursor powder microstructure and the EBM-fabricated components along with their residual tensile and hardness properties. Reticulated mesh components were also fabricated by EBM, and the Young’s modulus (dynamic stiffness) measured for these components with varying density (or porosity) using resonant frequency detection.

2. Experimental procedures

Rapidly solidified, gas atomized pure iron powder having sizes, size distribution, and microstructures as shown in Figs. 1–3 were processed in an upgraded, Arcam S-12 electron beam melting (EBM) system illustrated schematically in Fig. 4. Figs. 1(a) and 3(a) show the details of the precursor powder morphologies and size distribution, with an average particle size of 19 μm as shown in Fig. 3(a). Fig. 1(b) shows polished and etched particle cross-sections illustrating an irregular but equiaxed grain structure of roughly 3 μm diameter as illustrated in the magnified scanning electron microscope (SEM) image in Fig. 2. Although the powder crystal structure exhibited primarily α-Fe (bcc; a = 2.87 Å), a δ-Fe (1 1 0) (bcc; a = 3.0 Å) peak was observed by X-ray diffraction (XRD) analysis as indicated by the arrow in Fig. 3(b).

The EBM system (Fig. 4) upgrade consisted of an air-cooled electron gun (at G in Fig. 4), with an insulating space between the gun and the build chamber (below C in Fig. 4). The system also incorporated additional heat shielding to decrease heat loss during component fabrication. The electron optical system includes beam focus at F in Fig. 4 as well as beam deflection at D. The atomized powder (Fig. 1(a)) was loaded into cassettes (P) where the powder was gravity fed to the build table and raked into layers (at R in Fig. 4). The powder layer was preheated in successive electron beam scans at speeds of ~10^3 mm/s at low beam current to achieve temperatures around 300 °C. Preheating was followed by a melt scan.
at scan speeds of 200–300 mm/s at increased beam current. The system operates at a beam voltage of 60 kV in a vacuum of ~10⁻⁴ Torr.

In this research program, solid cylindrical components were fabricated using 1.5 cm diameter × 10 cm in length, along with a range of reticulated mesh structures using a dode-thin build element (a rhombic dodecahedron shape) using Materialise Software, embedded in a CAD program to direct the layer melting. This mesh element was selectively manipulated and expanded to produce open-cellular mesh components having different strut diameters (ranging from ~1 to 1.5 mm) and mesh dimensions to produce a range of densities. These mesh components measured nominally 2.3 cm × 2.3 cm × 3.5 cm height.

Standard tensile specimens were machined from the solid cylindrical components and tested at room temperature (22°C) at a strain rate of 10⁻³ s⁻¹. The elastic (Young’s) modulus of the reticulated mesh components were measured using a resonant frequency and damping analyzer, where the Young’s modulus (E) is proportional to the resonant frequency squared [4]. These measured Young’s moduli were then divided by the solid Young’s modulus determined from tensile testing to produce specific modulus (or stiffness) values (E/E₀), which were compared to corresponding, specific density values obtained by dividing the measured mesh component density by the measured, solid density (ρ/s). These were plotted on a log-log plot to examine the exponent(n) in the standard open-cellular structure (foam) equation: \( E/E_0 = (\rho/\rho_s)^n \) [5].

Both the solid and mesh component samples were sectioned and mounted, polished, and etched (with Kalling’s No. 2: 5 g CaCl₂ in 100 mL HCl and 100 mL ethanol) to observe the corresponding microstructures by optical metallography, and for microindentation (Vickers) hardness testing, using a 100 gf load. This included the initial, rapidly solidified powders as implicit in Fig. 1(b).

As illustrated in Figs. 1(a) and 2, powder samples and the EBM fabricated components were examined by SEM, using a Hitachi S-4800 field-emissions SEM operated at 20 kV accelerating potential. The SEM employed an EDX energy-dispersive X-ray spectrometer as well. SEM analysis also included observations of the fracture surfaces for tensile-tested specimens.

X-ray diffractometry (XRD) analysis was performed on the precursor powders (Fig. 3(b)) as well as the as-fabricated specimen sections cut from solid and mesh EBM components. In the solid, cylindrical specimens, XRD spectra were compared for horizontal and vertical plane sections perpendicular and parallel to the EBM build direction, respectively. A Bruker AXS-D8 Discover X-ray diffractometer system utilizing a Cu (Kα) X-ray source was employed in the XRD analyses.

Thin sections were cut from the solid EBM-fabricated cylindrical components oriented in the horizontal and vertical reference planes (perpendicular and parallel to the
build direction) as noted above. These sections were ground to a thickness of \(~200\mu m\) from which 3 mm discs were punched and mechanically dimpled to allow for the preparation of electron-transparent specimens for transmission electron microscopy (TEM). The punched, 3 mm discs were electroetched in a dual-jet electropolishing system using a solution consisting of 50 mL perchloric acid, 950 mL acetic acid solution at 35 V and a temperature of 10 \(^\circ\)C. The electron transparent iron specimen discs were observed in a Hitachi H9500 TEM operating at 300 kV accelerating potential; utilizing a goniometer-tilt stage.

3. Results and discussion

Fig. 5 shows a 3D-optical metallograph composite illustrating the microstructure for a typical solid component section, while Fig. 6 shows magnified image sections representing the horizontal (H) and vertical (V) reference planes. While the grains are more equiaxed and larger than those of the precursor powder (Fig. 1(b)), there is a homogeneous distribution of small, oriented inclusions within the grains. As illustrated in the corresponding XRD spectra representing the horizontal

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**Fig. 5** – 3D-optical metallograph composite section for EBM-fabricated Fe component. B denotes the build direction in Fig. 4. H and V denote the horizontal and vertical reference planes, respectively. The average grain size in the horizontal reference plane was 9 \(\mu m\) while in the vertical reference plane it was 16 \(\mu m\).
Fig. 6 – Magnified views for the horizontal (a) and vertical (V) reference plane microstructures in Fig. 5.

Fig. 7 – XRD spectra for (a) the horizontal and (b) the vertical reference planes corresponding to Figs. 5 and 6.

Fig. 8 – TEM bright-field image showing δ-Fe phase plates along α-Fe (100) planes noted by trace directions A = [101]; B = [110]. The operating reflection g = [011] in the selected-area electron diffraction pattern insert. Image is in the horizontal reference plane (H) in Fig. 5.

Fig. 9 – TEM bright-field image showing δ-Fe plates in α-Fe (1 1 0) planes in (1 1 0) grin surface orientation (selected-area electron diffraction pattern insert). g = [1 1 0].
Fig. 10 – TEM bright-field image showing $\delta$-Fe phase plates in $\alpha$-Fe (1 0 0) planes along trace directions $A = [0 0 2]$ and
$B = [1 1 0]$ (large letters) in a (1 1 0) surface orientation (SAED insert); in a vertical reference plane section (Fig. 5).
The small letters $A$ and $B$ in Fig. 10 represent $A = [1 1 1]$ and
$B = [1 1 1]$ directions parallel to screw dislocation lines. Dislocations in $\alpha$-Fe (1 1 0) planes.

and vertical reference planes in Figs. 5 and 6, shown in Fig. 7, the homogeneous inclusions appear to be non-equilibrium
$\delta$-phase which was observed in the precursor powder as illustrated in the XRD spectra in Fig. 3(b). This is confirmed in a
series of TEM, bright-field images representing several common crystallographic (zone axis) orientations in both the horizontal and vertical reference planes as shown in Figs. 8–13.

Fig. 8 illustrates a [1 0 0] zone orientation with 40 nm thick
$\delta$-phase, irregular plates coincident with (1 1 0) $\alpha$-Fe matrix
planes which intersect at $90^\circ$ and are normal ($90^\circ$) to the (1 0 0)
grain surface. The selected-area electron diffraction (SAED) pattern insert in Fig. 8 shows the [0 1 1] operating reflection
(g) while the $\delta$-phase trace directions shown at $A$ and $B$
correspond to [0 2 0] and [1 1 0], respectively. The TEM image in
Fig. 8 also shows a heavy dislocation structure. Fig. 9 shows a
[1 1 0] zone grain also in the horizontal reference plane (Fig. 5)
showing the irregular, oriented $\delta$-phase in the [0 0 2] direction,
coincident with the $\alpha$-Fe matrix (0 1 0) plane. The (0 1 0) planes
make an angle of $45^\circ$ with the $\alpha$-Fe (1 1 0) grain surface and
showing the projected $\delta$-phase contrast fringes corresponding
to the [1 1 0] operating reflection (g) in the SAED pattern
insert in Fig. 9.

Fig. 10 shows the same [1 1 0] zone axis $\alpha$-Fe grain orientation
as Fig. 9 but in a vertical reference plane section (Fig. 5).
However in Fig. 10, the $\delta$-phase plates exist on both the (0 1 0)
plane (as in Fig. 9) and the (0 0 1) plane, which is perpendicular
($90^\circ$) to the (1 1 0) grain surface plane; corresponding to the

Fig. 11 – TEM bright-field image showing an oblate $\delta$-phase plate in the $\alpha$-Fe (0 1 0) plane in a (1 1 0) oriented $\alpha$-Fe grain.
The $\alpha$-Fe grain is in the vertical reference plane (Fig. 5).

Fig. 12 – TEM bright-field image showing $\delta$-phase plates in
(1 1 0) planes along $A = [1 0 1]$ and $B = [0 1 1]$ trace directions
in a (1 1 1)-oriented $\alpha$-Fe grain (SAED pattern insert). The
(1 1 1) $\alpha$-Fe grain is in the vertical reference plane (Fig. 5).
crystal plane geometry in Fig. 8, where the (110) δ-phase plane was perpendicular to the (100) α-Fe grain surface plane. Large A and B show the corresponding [002] and [110] trace directions, respectively. In addition, small A and B shown at long (presumably) screw dislocation lines correspond to \(A = [11\overline{1}]\) and \(B = [11\overline{1}]\) directions. These would ideally represent (111) Burgers vectors in the (110) α-Fe slip planes; further confirming the characteristic crystallographic features noted for the δ-Fe phase plates. The plate thickness measured for the δ-phase coincident with the (001) plane along large B in Fig. 10 also average 40 nm thickness as already noted for Fig. 8. The dislocation images and δ-phase in Fig. 10 correspond to a [110] operating reflection (g) shown in the SAED pattern insert in Fig. 10.

Fig. 11 shows an oblate δ-phase platelet in the (010) α-Fe matrix plane which makes a 45° angle with the (110) grain surface plane. The corresponding fringe contrast arises from a [110] operating reflection as in Fig. 10.
Fig. 14 – SEM views for mesh strut structure. (a) Cross-section view through strut sowing loosely sintered surface particles. (b) Magnified view of strut surface structure showing complete melting under powder particles.

Fig. 12 shows a bright-field TEM image for δ-phase plates coincident with (110) α-Fe matrix planes which make an angle of 35° with the (111) α-Fe grain surface orientation indicated by the SAED pattern insert. The trace directions indicated by A and B in Fig. 12 correspond to [101] and [011], respectively. There is also a high dislocation density associated with the α-Fe matrix in Fig. 12.

Fig. 13 shows examples of seven different reticulated mesh components fabricated by EBM, corresponding to densities indicated. These examples are viewed at 90°, corresponding to square and hexagonal geometries on opposing faces. These are created by systematic rotation of the build element in the CAD model represented in Fig. 13(c). Fig. 13(a) also illustrates the variation in the strut diameters, which increase from left to right. Typical strut structures are illustrated in the SEM images in Fig. 14, which show relatively unmelted and partially sintered Fe powder particles in the surface, and composing several equivalent particle diameters in surface thickness. The strut interior solid portion is shown in cross-section in Fig. 14(a). Fig. 14(b) shows surface melt and additive-layer melt features.

Fig. 15 – Stress-strain diagram (a) and specific stiffness (E/Es) versus specific density (ρ/ρs) log-log plot for the reticulated mesh components represented in Fig. 13(a) and (b). The slope of the fitted line is 2.8.

Fig. 15(a) shows an example of the tensile, stress–strain response for the solid, cylindrical Fe components. The solid density was measured to be 7.51 g/cm³. In contrast to the theoretical density of 7.87 g/cm³, the measured density indicates a porosity of roughly 4%. In addition, the tensile data represented in Fig. 15(a) shows an upper yield point which, when averaged with other tensile data, was observed to be 170 MPa; and an ultimate tensile strength of 180 MPa. The Young’s modulus measured from the tensile data (as in Fig. 15(a)) was 116 GPa. With this modulus (Es = 116 GPa) and a solid density of 7.51 g/cm³ (ρs = 7.51 g/cm³), the specific stiffness (E/Es) and specific density (ρ/ρs) for the mesh components (Fig. 13(a) and (b)) were plotted as illustrated in Fig. 15(b), where the slope of
the fitted line was observed to be \(~2.8\). This is larger than the average \(n\equiv2\) characteristic of a range of other metal and alloy mesh and foam materials \[6,7\]. The significant portion of the mesh struts having poorly melted and partially sintered surface particles (Fig. 14) may have contributed to this variance in the specific stiffness versus specific density data represented in Fig. 15(b). In addition, the sample dimensions noted in Fig. 13 are at the edge of the dynamic measurement sensitivity. Consequently a combination of issues has contributed to the measured slope shown in Fig. 15(b).

It might also be noted that the measured Young’s modulus also reflects the well known effect of porosity, where for low porosity (4.6% measured in this study) \(E/E_s = 1–1.9P\), where \(P\) is the porosity \[8\]. Consequently the ideally dense solid would be 127 GPA.

Table 1 – Mechanical properties for EBM-fabricated Fe.

<table>
<thead>
<tr>
<th>Powder hardness* (GPa)</th>
<th>Mesh hardness* (GPa)</th>
<th>Solid hardness* (GPa)</th>
<th>Young’s modulus (GPa)</th>
<th>Yield strength (GPa)</th>
<th>UTS (GPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5</td>
<td>1.26</td>
<td>1.28</td>
<td>116</td>
<td>0.17</td>
<td>0.18</td>
<td>15</td>
</tr>
</tbody>
</table>

* Vickers, 100 gf load, 10 s dwell (20 measurements average).

![Fig. 16 – SEM views for fracture surface sections corresponding to tensile specimen failure (as shown in Fig. 15(a)).](image)

(a) Lower magnification. (b) Higher magnification image showing ductile dimple detail. Average dimple size is \(~0.4\mu m\). Magnification in (a) is the same as noted in (b).

Fig. 15(a) also illustrates an elongation of 15% while Fig. 16 illustrates the ductile dimple fracture surface microstructure characteristic of this moderate ductility. In addition, Fig. 15(a) shows, in addition to the upper yield point, discontinuous yielding evidence up to a strain (or elongation) of 0.05 (5%). This is probably related to the effect of the \(\delta\)-phase plates that are homogeneously distributed throughout the EBM-fabricated components as illustrated typically in Figs. 5 and 6. The dislocation substructures apparent in all of the TEM images (Figs. 8–12) will densify with increasing tensile straining and interact with the \(\delta\)-phase to create the discontinuous or irregular deformation straining implicit in the stress–strain diagram illustrated in Fig. 15(a).

The presence of the \(\delta\)-Fe platelets in the precursor, rapidly solidified powder, and their retention and even proliferation in the EBM-fabricated components would seem to be a characteristic of the rapid, non-equilibrium cooling associated both with the powder atomization and the layer-by-layer melt-solidification processing in EBM component building. Since the authors are unaware of any similar observations of retained, non-equilibrium \(\delta\)-Fe in \(\alpha\)-Fe, it is not possible to examine the associated and contributing thermokinetic phenomena in any detail. Nonetheless, the observations of \(\delta\)-Fe plates coincident with \(\{10\ 0\}\) and \(\{11\ 0\}\) \(\alpha\)-Fe matrix planes in Figs. 8–12 certainly present very novel circumstances which may necessitate further, more extensive study.

Table 1 summarizes the tensile data along with the microindentation hardness data measured in this study. It is of interest to note that the yield stress for the solid Fe components is roughly a factor 1/7.5 times the residual microindentation (Vickers) hardness. For many metals and alloys the yield stress is 1/3 the corresponding microindentation hardness.

Both the solid EBM-fabricated components and the mesh components were highly magnetic, as determined by exposing them to a strong rare-earth magnet.

4. Conclusions

Rapidly solidified, atomized pure iron powder having an average powder particle size of 19 \(\mu m\) was observed to contain meta-stable \(\delta\)-Fe (bcc; \(a\equiv3.0\) \(\AA\)) which was retained and enhanced in the EBM fabrication of solid and reticulated mesh components. EBM fabricated solid Fe components were observed by TEM to contain a homogeneous distribution of \(\delta\)-Fe phase plates measuring roughly 0.5 \(\mu m\) to 2 \(\mu m\) in length and \(~40\) \(nm\) thick, coincident with either \(\{10\ 0\}\) or \(\{11\ 0\}\) \(\alpha\)-Fe matrix planes. This appears to be the first such observation of \(\delta\)-Fe in and \(\alpha\)-Fe matrix.
Measurements of the dynamic stiffness (Young's modulus) for EBM-fabricated Fe mesh components allowed the specific stiffness \((E/\rho_s)\) to be plotted against specific density \((\rho/\rho_0)\) in a log-log plot where it was observed that for \((E/\rho_s) = (\rho/\rho_0)^n\), \(n = 2.8\). The solid density was measured to be 7.51 g/cm\(^3\) while the corresponding Young's modulus was measured from stress–strain plots to be 116 GPa. Tensile testing showed an upper yield point of 170 MPa with evidence of discontinuous yielding possibly due to the presence of the \(\delta\)-phase platelets. The tensile elongation was observed to be 15%. The initial powder microindentation (Vickers) hardness was 1.5 GPa in contrast to 1.26 GPa for the mesh components and 1.28 GPa for the solid components fabricated by EBM.

Conflicts of Interest

The authors declare no conflicts of interest.

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