

Full length article

Improving the mechanical properties of Fe – TiB₂ high modulus steels through controlled solidification processesH. Zhang, H. Springer^{*}, R. Aparicio-Fernández, D. Raabe

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ARTICLE INFO

Article history:

Received 4 July 2016

Received in revised form

25 July 2016

Accepted 28 July 2016

Keywords:

Steel

Young's modulus

Density

Ductility

Strength

Toughness

ABSTRACT

We investigated novel pathways to improve the mechanical properties of liquid metallurgy produced Fe – TiB₂ based high modulus steels (HMS) by controlled solidification kinetics and subsequent thermo-mechanical treatments. The solidification rate was varied by casting of hyper-eutectic alloys (20 vol% TiB₂) into moulds with differing internal thickness. Ingots between 5 and 40 mm thickness exhibited irregular particle microstructure consisting of sharp-edged coarse primary particles (increasingly clustered with slower solidification) and closely spaced irregular lamellae. Casting defects can be alleviated by hot rolling, but the mechanical properties remain unsatisfactory. Increasing the solidification rate results only at mould thicknesses of 4 mm and below in a significant refinement of the particle microstructure, necessitating liquid metal deposition techniques to utilise it for obtained improved mechanical performance of HMS. Decreasing the solidification rate causes density-induced floatation of the primary particles, which can be used in block-casting for the production of alloys consisting of small and spheroidised eutectic particles, exhibiting high ductility and superior toughness. Annealing just above the solidus-temperature allows the eutectic zones to liquefy and sink, leaving only primary TiB₂ particles behind in the top zone of the alloy. Despite the increased particle fraction up to 24 vol%, both strength, specific modulus and ductility are improved over standard processed HMS alloys with 20 vol% TiB₂.

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1. Introduction

Steels represent the most common and successful structural materials due to their unprecedented spectrum of mechanical and physical properties. Current steel design studies pursue multiple directions, for example for further improving corrosion [1] and wear resistance [2], high temperature stability [3] and energy absorption [4]. For lightweight design such as required in transportation systems, the major challenge lies in increasing strength and reducing the density (ρ). Only recently, also stiffness (i.e. the Young's modulus; E) is being considered as a design criterion, as an increased specific modulus (i.e. E/ρ) – above the typical value of about 26 GPa g⁻¹ cm³ for steels, aluminium (Al), magnesium (Mg) and titanium (Ti) alloys – offers utilising yet untapped potential in lightweight materials design [5]. For this purpose, so called high modulus steels (HMS) have been developed, i.e. iron (Fe) based metal-matrix-composites, which allow to blend lightweight and

stiff phases with strong, ductile, tough, and not the least cost effective steel matrices [6,7].

In this light, the Fe – Ti-diboride (TiB₂) system has received considerable attention. TiB₂ is not only very effective ($E \sim 565$ GPa, $\rho \sim 4.25$ g cm⁻³ [8]), but also allows for volume production of HMS via liquid metallurgical synthesis methods, as TiB₂ – unlike for example most oxides or nitrides – can be precipitated from a homogenous Fe – Ti – B melt in a pseudo-binary eutectic reaction [9] (Fig. 1a). Furthermore, interface analyses at the atomic scale revealed a strong interfacial cohesion between Fe and TiB₂ particles [10]. According to theoretical predictions, i.e. a rule of mixture for ρ and the Halpin-Tsai model for E [10], respectively, the specific modulus increases with the TiB₂ fraction to about 37 GPa g⁻¹ cm³ for 30 vol % TiB₂ (Fig. 1b). However, if the TiB₂ fraction rises above 12 vol% (~6.3 mol.%), the alloy is above the eutectic TiB₂ concentration (Fig. 1a), and as a consequence, mechanically unfavourable TiB₂-morphologies result; i.e. coarse particles stemming from primary solidification additional to the already sharp-edged lamellae from the eutectic decomposition [6,7]. Additionally, the TiB₂ particles are rather brittle (K_{IC} of TiB₂ ~ 5 MPa m^{1/2} [8]) and their

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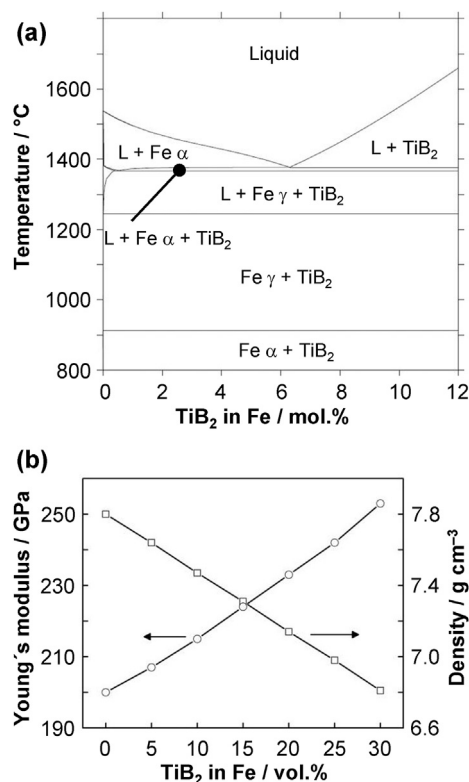


Fig. 1. (a) Phase diagram of the pseudo-binary Fe-TiB₂ system calculated with ThermoCalc. (b) Prediction of Young's modulus (Halpin-Tsai model [10]) and density (rule of mixture) of Fe for different volume fractions of TiB₂.

elevated stiffness induces stress concentration at the interface with the steel matrix. All this together causes pronounced embrittlement of the bulk material with increasing particle fractions, i.e. a deterioration of the mechanical performance with improvement of the physical properties.

One promising pathway to improve the ductility of HMS is to utilise alloying additions in order to modify the constitution and mechanical performance of the steel matrix, exceeding the properties of a pure Fe reference matrix (i.e. ferrite) towards stronger and more ductile multiphase microstructures well known from advanced high strength steel concepts [11,12]. The effect of the alloying additions on the particles' morphology is limited though [13], and thus the challenge remains to achieve a more mechanically compliant TiB₂ shape, size and dispersion. As the TiB₂ particles represent solidification products and are thermodynamically stable in the solid HMS, their morphology can not be significantly influenced through dissolving and re-precipitation processes, as it is commonly applied for example with alloy carbides in tool steels [14]. Thermo-mechanical treatments (TMT; i.e. hot and cold rolling) have been reported to have pronounced effects on the HMS microstructure, namely, inducing plastic deformation of the particles in case of hot rolling [15] as well as their fragmentation [16,17]. The latter phenomenon leads to embrittlement of the material and reduces its stiffness [17]. However, we recently demonstrated how TiB₂ particles can be effectively influenced by controlling the solidification rate of HMS melts, thereby refining the particles shape away from irregularly sharp-edges to spheres, as well as reducing their sizes by several orders of magnitude down to the nanometre range [6]. Especially the density-induced separation of primary and eutectic particles, observed for very slow cooling conditions [6], is extremely promising, as it can be utilised for the efficient production of large quantities of HMS under industrially controllable

conditions. While these processes can be readily combined with the aforementioned alloy design of the steel matrix in a later stage, their extent and thus the cost and associated processing efforts may be reduced to achieve HMS with superior physical and mechanical property profiles.

2. Objective

The objective of this work is to systematically study how the inverse relationship between mechanical and physical properties of Fe – TiB₂ based HMS can be overcome by tailored solidification processes. We aim at developing processing guidelines to obtain high particle fractions to increase the specific modulus without sacrificing the material's ductility and toughness, suitable for volume production and as the basis for the alloy design of HMS.

3. Materials and methods

3.1. Synthesis and processing

All alloys in this study were of the nominal composition Fe – 10.10 Ti – 3.86 B (wt.%; corresponding to a hypereutectic concentration with ~20 vol% TiB₂), and were prepared by melting pure metals in a vacuum induction furnace (VIM) under an argon (Ar) atmosphere. The Ti content was chosen about 17% over the stoichiometric amount required for TiB₂ (resulting in about 1.9 wt% Ti in the ferritic matrix) in order to suppress the formation of Fe-borides and to neutralise any inadvertently present carbon by the formation of Ti-carbides [19]. TiC carbides were found to precipitate in epitaxy with the basal plane of TiB₂ particles [10]. The solidification rate was varied by the internal thickness of rectangular water-cooled copper (Cu) moulds from 40 mm down to 1 mm (all moulds 40 mm wide), increasing the solidification rate with decreasing ingot thickness. As moulds with an internal thickness below 1 mm could not be filled reliably with liquid metal, even more rapid solidification was achieved by melt spinning: Charges of about 10 g, cut from the 10 mm thick VIM samples, were molten at 1600 °C in a boron nitride crucible and ejected through the slit-nozzle with an Ar pressure of 300 mbar onto a water-cooled Cu wheel rotating at a tangential velocity of 10 m s⁻¹, rendering ribbons of 100–500 μm thickness. The cooling rate for 20 mm thick VIM casts was measured to be about 10 K s⁻¹ [6], whereas during melt spinning ~10⁵ K s⁻¹ can be achieved [20]. Hot rolling followed by air cooling was performed on selected materials at 1100 °C to a final thickness of 2 and 6 mm, respectively.

In order to investigate the density-induced separation of primary and eutectic particles in more detail, samples from 10 mm thick VIM ingots were reheated to various temperatures between 1250 and 1640 °C. Critical temperatures of the alloy (e.g. transition, solidus and liquidus temperatures) and corresponding microstructures were determined by differential scanning calorimetry measurements (DSC; NETZSCH 404C) and differential thermal analysis (DTA; Setaram SETSYS-1750). DSC and DTA experiments were performed with cylindrical samples (3 mm diameter, 3 mm long), placed in alumina crucibles under Ar atmosphere, heated and cooled at 1 K min⁻¹ (no holding time at temperature). The first and second order transitions were revealed by the extrapolated peak-onset temperatures [21,22].

3.2. Characterisation and testing

Microstructures were characterised with a field emission scanning electron microscope (SEM; JEOL-6500F, operated at 15 kV) for imaging and electron backscatter diffraction analysis (EBSD; step size 50 nm, TSL OIM analysis software). ImageJ software was used

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