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In-situ metal matrix composite steels: Effect of alloying and annealing on morphology, structure and mechanical properties of TiB_2 particle containing high modulus steels

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We systematically study the morphology, size and dispersion of TiB_2 particles formed in-situ from Fe–Ti–B based melts, as well as their chemical composition, crystal structure and mechanical properties. The effects of 5 wt.% additions of Cr, Ni, Co, Mo, W, Mn, Al, Si, V, Ta, Nb and Zr, respectively, as well as additional annealing treatments, were investigated in order to derive guidelines for the knowledge based alloy design of steels with an increased stiffness/density ratio and sufficiently high ductility. All alloying elements were found to increase the size of the coarse primary TiB_2 particles, while Co led to the most homogeneous size distribution. The size of the eutectic TiB_2 constituents was decreased by all alloying additions except Ni, while their aspect ratio was little affected. No clear relation between chemical composition, crystal structure and mechanical properties of the particles could be observed. Annealing of the as-cast alloys slightly increased the size of the primary particles, but at the same time strongly spheroidised the eutectics. Additions of Co and Cr appear thus as the best starting point for designing novel in-situ high modulus metal matrix composite steels, while using Mn in concert with thermo-mechanical processing is most suited to adapt the matrix' microstructure and optimise the particle/matrix co-deformation processes.

1. Introduction

Weight reduction represents one of the major challenges for structural materials design. Respective key material properties are not only high strength in order to reduce the wall thickness and hence volume of the part, but also a low density (ρ) and simultaneously a high Young's modulus (E) for improved stiffness. Metal-matrix-composites (MMC's) are of special interest in this light, as they allow blending the property profiles of strong, ductile and tough metallic matrices with stiff and low-density ceramic particles [1,2].

Iron (Fe)-based MMC's, also termed high modulus steels (HMS), are especially attractive, as Fe not only exhibits a similar specific modulus (E/ρ) as f. e. aluminium (about 25 GPa g cm^{-3}), but additionally offers widely scalable mechanical properties due to its multitude of equilibrium and non-equilibrium phase transformations, low production costs and simple recyclability [3,4]. Titanium diboride (TiB_2) has received considerable attention as a respective particle material for HMS design, as it is very effective (E/ρ about 125 GPa g cm^{-3}), can form a strong interface with steel matrices, and, most importantly, can be synthesised in-situ from Fe–Ti–B melts or powder agglomerates in a pseudo-binary eutectic reaction [5–9]. While the liquid metallurgy production of such Fe–TiB_2 in-situ HMS is thus readily possible, the significantly reduced ductility of such composite materials remains a major challenge. A variety of effects was observed to entail brittleness of in-situ metal matrix composite steels: first, the large volume fraction of the ceramic phase, which is required to achieve the desired effect on E/ρ can lead to intrinsically brittle percolation paths through the material. Second, many of the ceramic particles are characterised by sharp-edged shapes which can promote damage initiation. Third, in situ formed ceramic particles in the steel matrix tend to coarsen during the synthesis which facilitates particle fracture upon loading [10–16].

While it was recently shown that the morphology and size of TiB_2 particles can be effectively influenced by tailoring the solidification kinetics [17], another promising pathway to increase the mechanical performance of novel HMS is to utilise alloying

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additions and thermal/thermo-mechanical processing: As is well established in steel design, alloying elements may be chosen to alter the constitution, phase transformation kinetics, transport coefficients, interface energies, interface cohesion and thus the resultant deformation processes of both, the particles and the metallic matrix [18–20]. In HMS, through some of the effects mentioned above, the alloying elements may also change the particle size and morphology, as f. e. shown in grey cast iron or Al–Si alloys, where the graphite and Si-particles, respectively, are drastically affected by even small additions of e. g. calcium or strontium [21–24]. Furthermore, also the intrinsic properties of the ceramic particles may be affected by possible incorporation of alloying elements within TiB_2. Thermal processing such as annealing treatments (possibly combined with mechanical processing via f. e. rolling) may enhance the associated elemental partitioning of the alloying elements and, most importantly, lead to significant changes not only of the matrix microstructure but also of the particle morphology and heterophase interface properties. Similar effects have been exploited for improving the toughness of high carbon steels through the spheroidisation of hyper-eutectoid cementite lamellae achieved by carefully tuned annealing parameters [3,4]. The combined effects of alloying elements and processing with the aim to alter matrix and particles can thus be systematically utilized to optimise the co-deformation processes occurring when mechanically loading the composite material, thus opening novel pathways for the design of HMS with superior profiles of their physical and mechanical properties.

2. Objective

The objective of this work is to systematically elucidate the effects associated with alloying additions and annealing treatments on the size, morphology, chemical composition, mechanical properties and crystal structure of TiB_2 particles formed in-situ during solidification of Fe–Ti–B based melts. The derived knowledge aims at contributing to the development of guidelines for the mechanism-based alloy design of HMS.

3. Materials and methods

3.1. Alloy production

All alloys presented in this study are of the base composition Fe–10.10 Ti–3.86 B (wt.%). This represents a hypereutectic composition according to the pseudo-binary Fe–TiB_2 phase diagram, corresponding to a TiB_2 fraction of about 20 vol.% [17]. The Ti/B ratio was chosen higher than the exact stoichiometric composition required for TiB_2 in order to suppress the formation of Fe borides [5]. Synthesis was performed by arc-melting 20 g charges of high purity (>99.99%) metals under an argon atmosphere (four times flipping them upside down and remelting for thoroughly homogenisation) on a water cooled copper plate, resulting in a cooling rate at a rate of about 5 K s^{-1} [17]. The following elements were added by 5 wt.% each to the above listed base composition: chromium (Cr), nickel (Ni), cobalt (Co), molybdenum (Mo), tungsten (W), manganese (Mn), aluminium (Al), silicon (Si), vanadium (V), tantalum (Ta), niobium (Nb) and zirconium (Zr). These elements were selected as they represent common alloying additions of steels and can be expected to interact with the TiB_2 formation [3,25,26].

While the emphasis of this study lies on the alloying influence on the resultant microstructures and mechanical properties in the as-cast state, the effects of additional heat treatments was also evaluated for some selected alloys. For this purpose, samples containing Al, Mn, Co, Cr and Mo, respectively, as well as the base alloy, were additionally annealed at 1100 °C for 24 h under argon followed by water quenching to room temperature.

3.2. Characterisation

Cross sections of the button shaped specimen were cut by spark erosion and prepared by grinding and polishing with standard metallographic techniques. The microstructures were investigated by scanning electron microscopy (SEM; Jeol JSM 6490) for imaging and electron backscatter diffraction (EBSD; Jeol JSM-6500F; TSL OIM analysis software 7.2.0). Chemical analysis was performed as qualitative mappings with energy dispersive x-ray spectroscopy (EDX; JSM 6490) in the SEM and quantitatively with an electron probe micro analyzer (EPMA; Jeol JXA-8100, acceleration voltage 15 kV, working distance of 10 mm) with at least 15 measurements each. The crystallographic structural analysis was performed by X-ray diffraction on a Seifert Type ID3003 using Co Kα radiation with a wavelength of 1.78897 $\times$ 10^{-10} m. Rietveld refinement in MAUD version 2.33 software was utilised to calculate the cell parameters of TiB_2, using the harmonic texture model to refine the texture. Transmission electron microscopy (TEM; Jeol-2200FS) was performed on samples of selected alloys prepared with a focused ion beam system (FIB; FEI Helios Nanolab 600i).

Morphology, fraction and size of particles were evaluated by image analysis using the ImageJ software package. We distinguish between particles stemming from primary solidification (coarse, polygonal) and eutectic decomposition (fine, lamellar). The analyses were performed on SEM backscatter electron contrast images at magnifications of 500× for primary and 1000× for eutectic particles, corresponding to areas of 51.3 $\times$ 10^3 and 13.35 $\times$ 10^3 μm^2, respectively.

The mechanical properties, i.e. hardness and reduced Young's modulus (E_r) of primary TiB_2 particles (i.e. those being large enough to be tested) and matrix were probed by nanoindentation using a Hysitron triboindenter and a Berkovich-type indenter at a load of 1000 μN. A minimum of 15 indents were placed in at least three differently oriented particles and matrix grains, respectively. The E_r values were derived from the slope of the load–displacement curve during unloading according to Olivier and Pharr [27].

Liquidus, eutectic and solidus temperatures were calculated by thermodynamic equilibrium calculations using ThermoCalc software and the TCFE7 B–Ti–Fe database with adapted ternary parameters in the liquid phase (supplied by ThermoCalc, Sweden).

All scatter shown for particle sizes and morphology, hardness, E_r and chemical concentrations represent minimum and maximum values.

4. Results

4.1. Morphology and size

Examples of typical characterisation results from the base alloy, i.e. without any alloying additions, are compiled for the as-cast state in Fig. 1. SEM micrographs at different magnifications (Fig. 1a) show the coarse primary TiB_2 particles in square, triangular and hexagonal shapes (top image) and eutectic TiB_2 constituents (bottom image) mostly in form of sharp-edged lamellas as well as in irregular ‘flower’ or star-like shapes. This deterioration from a strictly regular lamellar morphology as formed under equilibrium conditions can be expected in view of the accelerated solidification rate of the synthesis route chosen here [17]. The measured total particle fraction (primary plus eutectic) of 17.6 vol.% is slightly below the predicted value of 20%. EBSD analysis (Fig. 1b, both maps superimposed with image quality data in grey scale) showed no evidence of intermetallic compounds in the base alloy (phase map