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Demonstration of a strategy to create hybrid materials with stochastic 3D mesostructures

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ABSTRACT

A strategy to create hybrid materials with stochastic 3D mesostructures is proposed. The strategy entails the combination of commodity materials significantly different in terms of length-scale and their processing via induction sintering. Materials composed of a ferrous filaments mesostructure embedded in a sintered titanium microstructure are manufactured as representative example. The study shows that the presence of the random 3D mesostructure prevents the catastrophic fracture by impact of the hybrid materials.

1. Introduction

Materials are the backbone of other scientific innovations and development. There is an ever-growing demand on structural engineering materials to be stronger, tougher, or simply provide a wider range of conflicting mechanical performance. In most materials, the properties of strength and toughness are mutually exclusive [1] and homogeneous materials cannot fulfil such conflicting requirements [2]. Materials made of heterogeneous domains having different constitutive behaviours have thus been proposed to overcome the strength-ductility trade-off [2]. Heterogeneous-structured materials can be subdivided into two categories: intergranular (e.g., hierarchical grain structures) or intragranular (e.g., nano-twins) architectural heterogeneities. Microstructure hybridisation was also investigated to obtain better strength/ductility pairs [3].

All these approaches are confined to the microscale and, therefore, are not necessarily the best option as the mechanisms controlling mutually exclusive properties can originate from and operate at very different length-scale [1]. A comprehensive strategy to successfully address conflicting properties would combine materials at different length-scales [2], entailing the combination of two or more existing materials to allow superimposition of their complementary and synergistic properties to create hybrid materials [4]. Although some hybrid metallic structures are available (e.g. sandwich panels), the production of tailored hybrid materials is still challenged by the need of significantly changing conventional manufacturing methods or developing new processing routes [4,5].

An interesting approach to manufacture hybrid materials considering the incorporation of a mesostructure has been proposed by Martin et al. [6] where an electron beam melted octet-truss lattice made out of Ti-6Al-4V alloy was embedded into a matrix made out of pure Ti using spark plasma sintering. Martin et al. [6] proved that the technique can produce materials with good bonding; however, the manufacturing of costly purposely-designed lattices along with their mm size limitation can be drawbacks.

2. Method

The present work investigates the approach of manufacturing hybrid materials with stochastic 3D mesostructures, conversely to have controlled 3D structures [6] generally obtained via additive manufacturing techniques [7], make use of pairs of commodity inorganic materials (Fig. 1). The aim is to prove the viability of this strategy to create hybrid materials and quantify the performance of a representative example. Specifically, in this instance a low cost HDH (hydride-dehydride) Ti powder (Fig. 1a) from Goodfellow Ltd. and galvanised filaments (Fig. 1b) from Rapid-AB were processed via the simple press and sinter powder metallurgy route to create hybrid materials with an internal mesostructure.

The Ti powder has irregular morphology (Fig. 1a), which makes it ideal to be shaped via uniaxial pressing. Two-type (i.e., C-shaped or elongated with double convex-bent edges (Fig. 1b)) of Zn-coated (10 μm) galvanised steel filaments were used to generate 3D stochastic structures (Fig. 1c). Random self-sustaining structures were achieved by pouring the filaments (approximately 25 g) inside a 56 mm cylindrical die, which was then fully covered with Ti powder (approximate ratio of

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3:1). Warm compaction was performed uniaxially at 230 °C applying 600 MPa. It is worth mentioning that if plastic deformation of the 3D random mesostructure wants to be prevented, the compaction pressure can be tailored accordingly. Green samples were consolidated via induction sintering (Fig. 1d) at 900 ± 10 °C under Ar using a Cu induction coil (∅90 mm) composed of 7 turns was powdered by an Inductotherm Power Track 15–96 unit set at ~ 10 kW [8]. The total sintering cycle (heating to temperature and cooling) was approx. 5 min [9].

The samples for the different characterisations were cut via electoral discharge machining. The relative density was obtained either via mass/volume ratio (ρ_g: green density) or Archimedes' principle measurements (ρ_c: consolidated) divided by the theoretical density. Microstructural analysis on etched samples (Kroll solution) as well as XRD (30-80°, 0.013° scanning step) were performed for phases identification. Unnotched Charpy impact tests (55 mm x 5 mm x 5 mm,), $HV_{0.1}$ microhardness (LECO LM-700), and tensile properties (dog-bone 2 mm x 2 mm x 20 mm, strain rate of $5{\cdot}10^{-3}$ s^{-1}, Instron 33R4204) were quantified. For each condition, a minimum of three measurements were performed.

3. Results and discussion

The microstructure of the hybrid materials is composed of the micrometric α-Ti grains and millimetric randomly distributed cross-sections of the filaments where the ferrous cores of the galvanised filaments and their Zn coating are clearly discernible (Fig. 2). The short sintering cycle guarantees that the 3D stochastic mesostructure is maintained but some dissolution of the filaments in the matrix occurs. Additionally, irregular and interconnected residual porosity is present due to the short sintering cycle and low temperature. The type of 3D stochastic mesostructure (i.e. filament geometry) affects more the compressibility (ρ_g: 76.7–82.3%) rather than the final relative density (ρ_c: 85.4–86.2%) where higher values are achieved with the double bent filaments. However, no significant differences are found during the microstructural analysis due to the stochastic distribution of the mesostructure.

Brittle fracture is the failure mode of the hybrid materials to the application of loads at high strain rates (i.e. impact). However, the presence of the 3D stochastic mesostructure prevents the complete catastrophic separation during impact (Fig. 3 insets). The mesostructure provides an extrinsic toughening mechanism via crack bridging, typical of biomimetic synthetic ceramics [1], by means of uncut 3D mesostructure elements which are effectively preventing fracture separation. The presence of residual porosity with irregular morphology in the hybrid materials (Fig. 2) can also affect the fracture toughness as pore size, shape, and distribution can create a preferential crack growth pathway. However, the difference in microhardness (Fig. 3b) between the microstructural features indicates that they have a strong influence on the impact strength.

The shape of the tensile stress–strain curves (Fig. 3c) reinforces the fact that the stochastic 3D mesostructure prevents the sudden catastrophic failure of the hybrid materials (i.e., progressive decrement of the strength with the increase of the elongation after peaking). It is worth mentioning that the preparation of the samples through cutting for mechanical testing (both impact and tensile) leaves some of the elements of the stochastic 3D structure cut at different locations along their geometry facing the surface of the testing samples (Fig. 3 insets). This clearly affects the values of the measured mechanical behaviour presented as the exposed cut filaments are regions were crack initiation is facilitated. It is also worth noticing that the generation of a 3D stochastic mesostructure can also be used to change the intrinsic stiffness of the hybrid materials, which is significantly lower (35.8 ± 9.3 GPa) when using double bent filaments compared to C-shaped (69.5 ± 7.8 GPa), despite being composed of the same commodity materials.

The mechanical response is better understood by analysing the interaction between the elements of the 3D stochastic mesostructure and the matrix induced by the consolidation process. The ferrous core of the galvanised filament (~0.5 mm, vertical cross-section, Fig. 4a) mainly remains intact, meaning that there is no direct interaction with the matrix. Line scan (Fig. 4d) and elemental mapping (Fig. 4b and c) show a clear distinct distribution of Fe and Ti in the core of the filament (which is fully dense) and in the matrix (which is porous). However, both elements as well as Zn are present in the original coating layer of the ferrous filaments. High magnification micrographs reveal that the uniform and homogeneous Zn coating obtained via hot dipping of the ferrous filaments transforms into a layer characterised by a eutectic-like structure layer of 118 ± 30 μm in thickness (Fig. 4e). Fe and Zn are both β eutectoid stabilisers in Ti and have extremely low/nil solid solubility in α-Ti. Due to the sintering cycle used, Fe and Zn mainly interact with α-Ti. The eutectoid-like structure is composed of TiFe and the Γ phase [10] as per the Ti-Fe-Zn phase diagram [11]. The presence of the TiFe intermetallic compound, along with pure Fe and α-Ti, was confirmed via XRD (Fig. 4e).

Microhardness values (Fig. 3b) further confirm that the failure of the hybrid materials is primarily due to the formation of intermetallic compounds at the interface between the matrix and the mesostructure. As the elements of the mesostructure have lower hardness, and thus expected higher ductility, avoidance/tailoring of the interface could be leveraged to create hybrid materials composed of soft and hard domains permitting to overcome the strength-ductility trade-off [2]. Therefore, engineering the presence of the eutectoid structure at the interface between the microstructure and the mesostructure, which provides an easy pathway for the crack to propagate during impact, will allow achieving more significant benefits in terms of catastrophic failure prevention in hybrid materials with stochastic 3D mesostructures through the generation of toughness in the material without modifying its ductility.

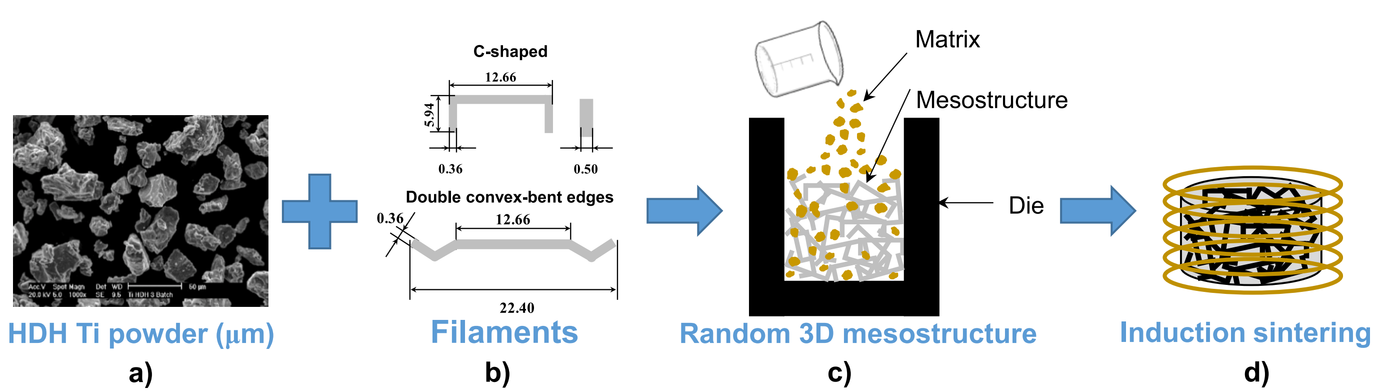

Fig. 1. Pathway for manufacturing hybrid materials: a) raw powder, b) filaments, c) creation of the stochastic 3D mesostructure, and d) consolidation via induction sintering.

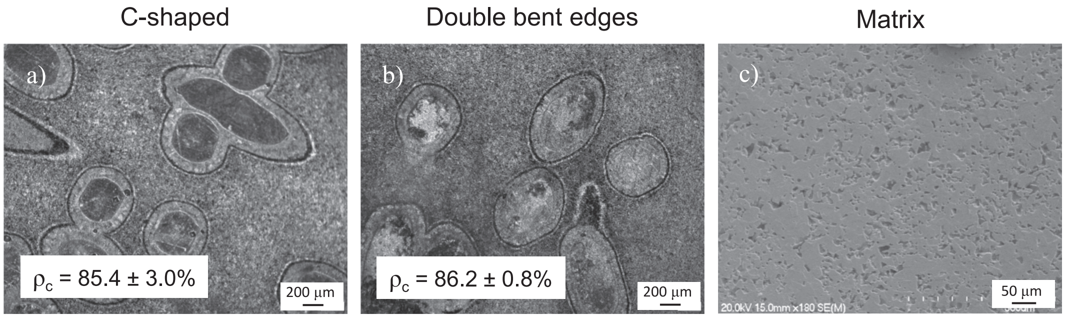

Fig. 2. Microstructural analysis: a) hybrid material with C-shaped filaments, b) hybrid material with elongated filaments with double convex-bent edges, and c) detail of the matrix.

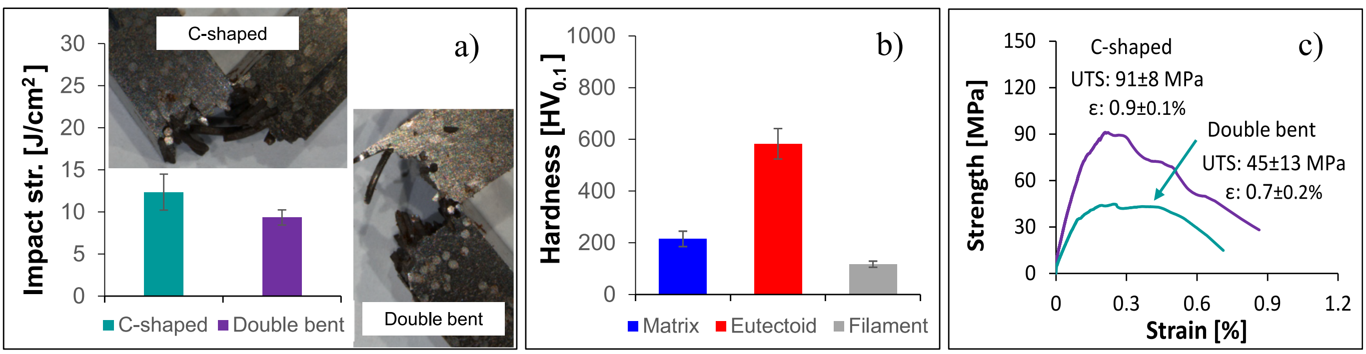

Fig. 3. Mechanical properties: a) impact strength and fracture surface as inset, b) microhardness, and c) representative stress–strain curves.

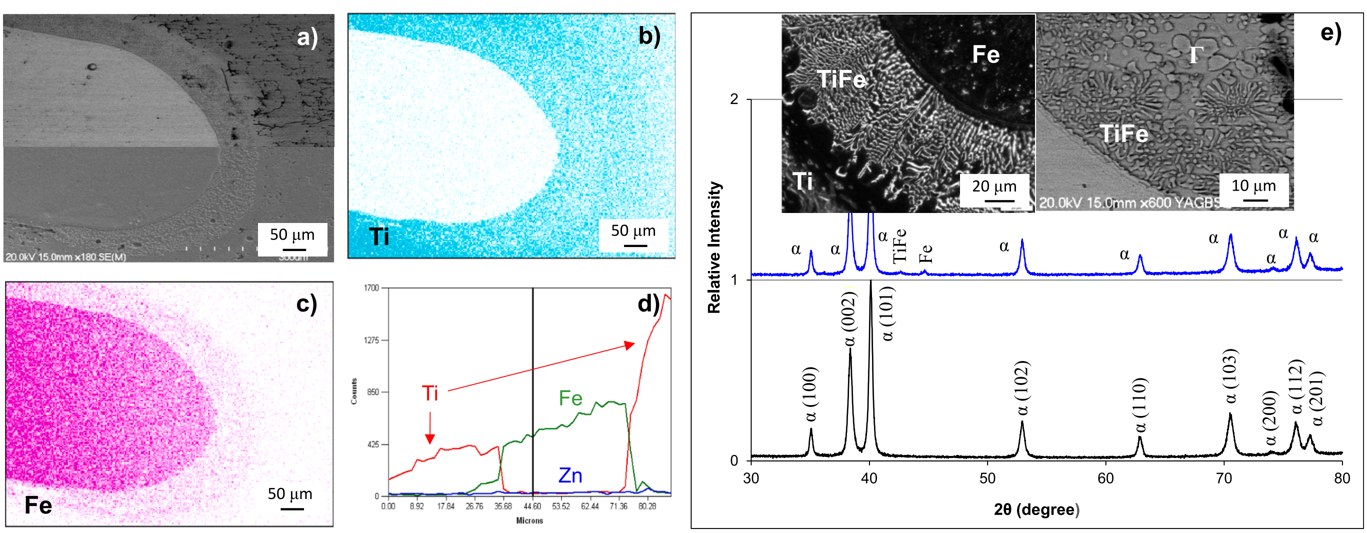

Fig. 4. Interface analysis: a-c) SE/BSE micrograph and elemental maps, d) EDS line scan, and e) XRD patterns and detail of the phases of the eutectoid structure.

4. Concluding remark

Via the representative example of combining Ti with galvanised filaments with two different geometries we demonstrate that the combination of commodity materials and smart manufacturing is a promising method to create hybrid materials even though improvement of the mechanical behaviour requires further optimisation. It is inferred that through this approach random 3D structures of strong/weak, hard/soft, tough/brittle materials can be achieved via the optimised selection of the starting commodity materials.

CRediT authorship contribution statement

A. Hussein: Methodology, Investigation. **F. Yang:** Methodology. **L. Bolzoni:** Writing – review & editing, Methodology, Investigation, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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