Experimental analysis of damage of Fe-TiB$_2$ metal matrix composites under complex loading

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Abstract
Experimental analysis of damage of new steel-based composites submitted to complex loading has been investigated by means of SEM-EBSD for in-situ four-point bending tests and monotonic and reverse simple shear tests. The TiB$_2$ particles show sharp morphological and crystallographic texture. Both matrix grain sizes effect and particle reinforcement are noticeable on the overall mechanical behavior. Damage occurs mainly by particles fracture and the matrix-particle interface remains very resistant even after large plastic strain. Automatic image processing permitted to quantify damage evolution with strain and with strain path. Damage was shown to increase with matrix strengthening, and was less important on reverse loading than monotinous loading.

Mots clefs: composite à matrice métallique, TiB$_2$, flexion in situ, cisaillement simple, endommagement

1 Introduction
Metal–matrix composites (MMC) with ceramic particle reinforcement have been developed as structural materials due to their good stiffness to weight ratio [1]. However, the presence of the second hard phase decreases the composite ductility [2] and involves damage occurrence, which limits the structural applications of such composites. Most studies carried on MMC’s damage focuses on Al based systems and deal with the effect of reinforcement size and shape [3, 4] and/or volume fraction [5-7]. For Fe-TiB$_2$ systems most authors focus on the manufacturing process and microstructural characterization [8, 9] . So far, to the author’s knowledge, the damage mechanisms of Fe-TiB$_2$ system under complex loading is not documented. The present study focus on a novel in situ cast iron based metal matrix composite reinforced by 11% TiB$_2$ in order to analyze overall statistically representative features of damage under complex loading and up to large plastic deformation, with a particular attention to matrix grain size and strain path change effect on damage evolution.

2 Materials and Experimental procedure

2.1 Materials
The Fe-TiB$_2$ composites obtained by in situ eutectic solidification and hot rolled sheets around 3 mm thick were provided by ArcelorMittal. They are made of ferritic steel reinforced by about 11% volume fraction of
TiB2 ceramic particles. Two composites with the same particles characteristics and different matrix grain sizes were considered in this work: small-grained (SG) and coarse-grained (BG) matrix composites with mean grain size 3µm and 17µm respectively. Unreinforced coarse-grained ferritic steel has also been used as reference material.

2.2 Mechanical tests

Four-point bending tests were performed on the CG composite using a home-made device mounted on a miniaturized tensile machine, with a 3000N load cell capacity. Parallelepipedic specimens, with dimensions given in Fig.1 (a), were cut off along the rolling direction (RD) or the transverse direction (TD) in the sheet plane. The bending plane (1, 3), as shown in Fig1 (a), corresponds to the (RD, ND) plane for the longitudinal sample and to the (TD, ND) plane for the transverse one. Bending was obtained by applying displacement in direction 3. The samples cross-sections were mechanically polished before testing. Monotinous and reverse simple shear tests along TD were carried with a new home-made shear device suited for thick sheets, mounted on a tensile testing machine, as for previous devices [10]. Parallelepipedic samples were cut off by spark machining. Samples dimensions were L*w*t, with L=38 mm, w=23 mm and t is the sheet thickness. The sheared zone dimensions were L*b*t (Fig 1b) with b=2.5 mm. The shear stress is given by \( \tau = F/(L\cdot t) \), where F is the force measured with the load cell of the tensile machine. The shear strain \( \gamma \) is given by \( \gamma = \tan \theta \). The tests were controlled by video-extensometry with constant imposed shear rate \( \dot{\gamma} = 1.732 \times 10^{-3} \text{s}^{-1} \). More details about simple shear tests are available elsewhere [10]. The reference frame for the tests was (SD, NS, ND), with SD=Shear Direction, NS= Normal to the Shear plane, ND=Normal Direction to the sheet. Assuming material isotropy and strain homogeneity throughout the sheared zone, the equivalent strain \( \varepsilon = \gamma / \sqrt{3} \) and the von Mises equivalent stress \( \sigma = \tau \sqrt{3} \) were used. Monotonous shear tests were conducted up to equivalent strain \( \varepsilon_1=3\% \), \( \varepsilon_2=10\% \) and \( \varepsilon_3=30\% \). Strain reversal tests were conducted with \( \varepsilon_2 \) forward strain, up to \( \varepsilon_3 \) equivalent cumulated strain.

2.3 Microscopy and image processing

FEG-SEM investigations and EBSD measurements required careful mechanical polishing involving specific diamond grinding discs and commercial colloidal silica suspension. EBSD scan steps have ranged from 0.05 to 0.25µm for SG and CG composites respectively. OIM\textsuperscript{TM} software was used for data processing. Image processing procedures using Matlab were developed for automatic detection of cracked particles on SEM micrographs in order to quantify damage of the composites after mechanical straining.

3 Results

3.1 Initial microstructure

The typical microstructure of Fe-TiB2 composite in the longitudinal plane is shown on the SEM micrograph in Fig.2a, with the corresponding distribution of the reinforcement particles size (Fig.2b). Two types of particles are encountered in the ferritic matrix, with a clear preferred orientation along the rolling direction: coarse hexagonal prismatic particles with 14µm mean size, and small particles exhibiting mainly round...
corners and various shapes. Small particles are 2µm mean size, more numerous and cover about 65% of total particles volume fraction. Approximately 3% of the particles were broken in the initial microstructure, due to previous damage during manufacturing. EBSD analysis of the material shows the sharp crystallographic texture of the particles, exhibiting c-axis mainly oriented along the rolling direction (Fig. 3a), whereas the matrix is characterized by a weak texture and a large spreading of grain size (Fig3b).

FIG. 2 – (a) SEM micrograph of as-received sample Fe–TiB$_2$ on the longitudinal plane. The black phase corresponds to TiB$_2$ particles; (b) Distribution of TiB$_2$ particle sizes

FIG. 3 – Inverse pole figure (IPF) map of the RD for (a) the TiB$_2$ particles and (b) the ferritic matrix.

### 3.2 Simple shear global behavior

The equivalent stress–equivalent von Mises strain curves for the Fe-TiB$_2$ composites and the unreinforced base alloy in monotonic and reverse simple shear till macroscopic failure are shown in Fig.4 (a) and (b). Composite matrix grain size refinement resulted in a strengthening increase of the composite, when comparing SG and CG composites response as shown in Fig. 4 (a) and (c), due to Hall–Petch strengthening mechanisms. Adding ceramic particles to the base alloy material resulted in an increase in strength due to load transfer from the matrix to the higher stiffness reinforcement particle and a decrease in ductility. Fig4 (b) indicates that macroscopic failure occurs later in reverse shear.

FIG. 4 – Equivalent stress vs equivalent von Mises strain curves for simple shear tests: (a) monotonic shear up to macroscopic failure of Fe-TiB2composites and the unreinforced alloy, (b) comparison of monotonic and reverse loading up to macroscopic failure of CG composite (c) reverse shear with €=10% prestrain up to €=30%
3.3 Damage features under complex loading

3.3.1 Observations of damage development during four-points bending
Prior to loading, some process-induced cracked TiB2 particles were identified (fig.4a). During bending tests damage initiates for both specimens in zones close to the outer fiber for global strain around 3.7% and occurs mainly by both small and large particles cracking (fig.4b). Cracks appear in a direction almost perpendicular to direction 1, which is consistent with tensile loading in this zone, and crack opening increases with plastic deformation.

![Image](a)(b)(c)

FIG. 5 – Sequences of micrographs of a representative area at 100µm from the outer surface of the transverse specimen during four point-bending. (a) \( \varepsilon = 0 \) (b) \( \varepsilon = 3.7\% \) (c) \( \varepsilon = 8\% \). Black arrows indicate small particles cracked by straining. Direction 1 and TD are horizontal.

3.3.2 Statistically representative damage under simple shear

3.3.2.1 General features
As in bending, damage mechanisms under simple shear involve mainly particle fracture in opening mode. Significant damage has been detected from \( \varepsilon = 10\% \) for both SG and BG composites. Particles-matrix interface remains resistant even after high matrix plastic deformation (fig. 6). This result is consistent with in a recent study at atomic level [11], which strongly support the good interfacial cohesion assumed for this novel steel-based composite. In reverse shear, and after cumulated strain \( \varepsilon = 30\% \), crack seems to be less open and more fragmented (fig 6.b), compared to monotonic shear for the same strain value (fig 6.a). Cracks are found in mono and polycrystalline particles. Statistics on misorientations profiles across cracks in monocrystalline and polycrystalline cracked particles (fig.7) indicates that while fractures are more frequent in polycrystalline particles, crack does not systematically follow TiB2 grain boundaries, so that fracture appear often intragranular in polycrystalline particles.

![Image](a)(b)

FIG. 6 - Damage aspect after \( \varepsilon = 30\% \). (a) monotonous shear (b) reverse shear with prestrain \( \varepsilon = 10\% \)
3.3.2.2 Quantitative analysis

- **Matrix grain size effect on damage under monotonous shear**
  Using automatic image processing the number of cracked particles was identified for both SG and BG materials, after $\varepsilon=3\%$, 10\% and 30\%. The total number of cracked particles is strain dependent as shown in fig.8, and increases with strain, in monotonous loading. While damage is similar in SG and BG materials until $\varepsilon=10\%$, the number of broken particles is much larger in SG materials at higher strain. It is worth noting that upon $\varepsilon=3\%$ the number fraction of cracked particles is almost equal to that identified in the initial state for both materials.

- **Strain path change effect on damage**
  As expected, quantitative analysis highlights greater damage upon monotonic shear straining, compared to that in reverse loading for the same cumulative equivalent strain amount (Fig.8) for the two studied composites (SG and CG). However, after strain reversal with $\varepsilon=10\%$, SG and BG composites exhibit similar damage quantities. Hence, matrix grain size effect on damage, sharp in monotonous shear as seen previously, is low in reverse shear.

![FIG. 8 – Effect of matrix grain size and strain path change on the number fraction of cracked particles as a function of the cumulated equivalent strain. Filled symbols for monotonous shear, open symbol for reverse shear.](image)

4 Conclusion

Initial and deformed microstructures under bending and shear tests have been investigated by SEM-EBSD for a novel iron based MMC reinforced by 11\% TIB2 particles. The main features of damage are:
- Damage occurs by particles cracking in opening mode, even during simple shear tests up to large plastic strain.
- Particle–matrix interfaces appear to be strong enough to resist decohesion even after 30\% equivalent strain.
- Damage is more frequent in polycrystalline particles and seems to be mostly of intragranular character.
- Quantitative analysis in simple shear shows that damage is more important in SG composite, in monotonous shear. In reverse shear damage is less important compared to the same monotonous shear strain amount, for both materials.

The results obtained in this study are a good basis for confrontation with modeling in progress.

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**References**