Data Article

Data related to spectrum analyzes for phases identification, microstructure and mechanical properties of additive manufactured Ti6Al4V reinforced with nano Yttria stabilized zirconia

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Ti6Al4V reinforced by nano yttria-stabilized zirconia (nYSZ) addition parts were manufactured via selective laser additive manufacturing. A research paper entitled “Effect of nano-yttria stabilized zirconia addition on the microstructure and mechanical properties of Ti6Al4V parts manufactured by selective laser melting” [1] discuss the microstructure and mechanical properties relationships. This data paper presents the analytical elements used to perform Rietveld refinement using X-ray diffraction data (XRD) obtained on manufactured parts in order to uncover microstructure characteristics (phase, crystallographic texture, lattice parameters, crystallite size, etc.). The XRD data are complemented by grains size/shape estimation and description obtained by Electron Backscattered Diffraction (EBSD).

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1. Data description

Ti6Al4V parts reinforced by nano yttria-stabilized zirconia (nYSZ) addition (see Table 1) were manufactured via SLM process. The discussion on the microstructure evolution and the related mechanical characteristics are presented in Ref. [1]. The data provided here, in support of the associated research paper were obtained by means of experimental investigation tools and techniques comprising spectrum analysis of powders and of manufactured parts by MAUD [2]. Raw and fitted spectrums are shown in Fig. 1 (Raw and fitted data are presented in Table A, supplementary file) in addition to texture analysis of α and β phases by LaboTex [3] (using XRD) as observed in Figs. 2 and 3, respectively (Raw data are presented in Table B in supplementary file.). Also, powder size distribution was estimated and shown in Fig. 4. In addition, Figs. 5–7 show the method used for grain size
calculation along with their aspect ratio and distribution, respectively. Using Electron Backscattered Diffraction experiments (EBSD) (parameters found in Table 2). Data processing was conducted by use of the Orientation Imaging Microscopy (OIM) software, version 7. Further, the data file describes how to process raw (uncorrected from the machine stiffness) compression tests data into a true stress - true plastic strain plots following an equation found in Ref. [5] and using raw data presented in Table C of the supplementary file.

2. Experimental design, materials, and methods

Four powder batches were prepared (Table 1) from which cubic-shaped parts were made (10 × 10 × 10 mm³) using selective laser melting (SLM) printer (SLM-125HL, SLM Solution, Germany). The following SLM melting process parameters were used: power 200 W; exposure 100 μs; hatch spacing 80 μm; point distance 65 μm; layer thickness 30 μm. Specimens were then extracted from each part for phase analysis using XRD, MAUD for Rietveld refinement analysis and LaboTex for texture analysis. Since the as-received Ti6Al4V powder possesses the same average particle size in all batches, the powder particle size distribution was measured on only one powder mixture, namely Bach#4 (Table 1). The particle size distribution was computed using images obtained by SEM as input, and a plot was generated by use of ImageJ software. As for the SLMed parts, the average grain size was measured by use of the OIM software, version 7 [4] through the exploitation of EBSD data. Compression tests were performed on all materials at room temperature, and at a quasi-static strain rate of ~10⁻³ s⁻¹. Detailed discussion of the data is given in Ref. [1].

2.1. X-ray diffraction analysis (XRD)

Experimental data were acquired on two INEL-THERMO FISHER SCIENTIFIC diffractometers. A two axes one—in asymmetric mode—to collect data for each phase analysis, and a four-circles one to collect data for crystallographic texture analysis of the two-phase bulk samples. Spectra Rietveld refinement analysis was performed via MAUD [2] software on both the as received Ti6Al4V powder and a mixture of Ti6Al4V and 2.5 wt% of nYSZ. In addition, refinements were also carried out on all fabricated parts presented in Table 1. For phase identification, Powder Diffraction Files (PDF) entry 04-002-8708 and 04-019-3251 of α and β phases respectively were used. PDF files of Tetragonal and Monoclinic zirconia were also provided to identify zirconia phases. Fig. 1 represents the typical Rietveld analysis via MAUD for all materials. The associated raw data are supplied in Table A (see the supplementary file). As examples, Figs. 2 and 3 represent the crystallographic texture analysis for Ti6Al4V-1nYSZ HT and Ti6Al4V-2.5nYSZ HT bulk samples that were extracted from the analysis of the raw data supplied in Table B (see the supplementary file). Texture effects were taken into account to determine α and β phases volume fractions using MAUD software.

2.2. Particles size distribution

The particles size distribution was measured using SEM image of a powder mixture of Ti6Al4V and 2.5 wt% of nYSZ, and then by using ImageJ software. The corresponding distribution is shown in Fig. 4.

### Table 1

| Powder batch data. For the heat treatment details, see Ref. [1]. |
|---------------------------------------------------------------|
| Ti6Al4V (wt%) | nYSZ (wt%) | Total weight (kg) | Abbreviation after melting | Heat treatment |
|----------------|-----------|-------------------|----------------------------|----------------|
| Batch#1        | 100       | 0                 | 5                          | Ti6Al4V        | No            |
| Batch#2        | 100       | 0                 | 5                          | Ti6Al4V HT     | Yes           |
| Batch#3        | 99        | 1                 | 5                          | Ti6Al4V-1nYSZ HT| Yes           |
| Batch#4        | 97.5      | 2.5               | 5                          | Ti6Al4V-2.5nYSZ HT| Yes           |
Fig. 1. Rietveld refinement analysis of (a) mixed powder (Ti6Al4V+ 2.5 wt% of nYSZ), (b) As-received Ti6Al4V powder, (c) As-built Ti6Al4V, (d) Ti6Al4V HT, (e) Ti6Al4V-1nYSZ HT, (f) Ti6Al4V-2.5nYSZ HT, and their corresponding zooms (g, h, i, j, k, l), respectively.
Fig. 2. The XRD crystallographic texture analysis of α phase for (a) Ti6Al4V-1nYSZ HT and (b) Ti6Al4V-2.5nYSZ HT bulk samples (via LaboTex software).

Fig. 3. The XRD crystallographic texture analysis of the β phase for (a) Ti6Al4V-1nYSZ HT and (b) Ti6Al4V-2.5nYSZ HT (via LaboTex software).
**Fig. 4.** Powder mixture size distribution.

**Fig. 5.** Ellipse fitted by OIM for grain size estimation [4].

**Fig. 6.** Evolution of the aspect ratio (length divided by width) of z lamellar grains of the melted parts.
2.3. EBSD analysis

EBSD experiments were performed following conventional polishing procedures described in Ref. [1]. The EBSD parameters used for the experiments are listed in Table 2. In addition, OIM analysis was carried out to investigate the effect of nYSZ addition, especially on the grain size of the α phase. No data cleaning procedure was applied. In order to estimate the grain size, grain shape method was used, this method consists of fitting an ellipse to points making up grain as shown in Fig. 5. By doing so, the evolution of the width and length of α grains is estimated. An aspect ratio curve was generated using the ratio of length and width of α grains of each material as shown in Fig. 6. For an ellipse-shaped grain,

| Table 2 | SEM acquisition setting. |
|---------|-------------------------|
| Step size | 0.08 µm |
| Magnification | 300× |
| Scan size | 115 × 115 µm² |

Fig. 7. Grain size map of α grains extracted from EBSD analysis. a, b, c and d correspond to Ti6Al4V, Ti6Al4V HT, Ti6Al4V-1nYSZ HT and Ti6Al4V-2.5nYSZ HT melted parts, respectively. The insets on each figure represent the length distribution. In each case, the minimum and maximum lengths of α grains are indicated by the X in the inserts.
the aspect ratio is defined as the ratio of the major axis to the minor axis. The aspect ratio data and average grain length size were extracted from EBSD grain size map shown in Fig. 7. Both Figs. 6 and 7 indicate a general trend toward a quasi-linear decrease of the aspect ratio, which reached the lowest value for the highly reinforced alloy.

2.4. Compression data

Compression tests were performed on the bulk parts according to ASTM E9. As no extensometer could be used, and because the machine stiffness was not available, the mechanical behavior was best described in terms of true stress versus true-plastic strain plots. Such plots were obtained from the raw data supplied in Table C (see the supplementary file). For each bulk sample, the compression true stress and true strain were first computed. Then, the truth plastic strain was estimated by use of the following expression [5]:

$$\varepsilon_p = \varepsilon - \left(\frac{b}{a}\right) - \left(\frac{\sigma}{a}\right),$$

where b and a are two constants taken from a linear function of the elastic part of the true stress-strain curve, \(\sigma\) and \(\varepsilon\) are the true stress and true strain, respectively. Such a description for the as-built Ti6Al4V can be plotted using compression test raw data provided in Table C (see the supplementary file). The details of the mechanical properties for all the bulk samples investigated (Table 1) are given in Ref. [1].

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Conflict of 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.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2020.105249.

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