High-Speed Nanoindentation Mapping of Additive Manufactured Titanium Alloys for Aerospace Application

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Abstract. High-speed nanoindentation mapping was carried out using a nanoindenter (iMicro, KLA Tencor, USA) using the method of Nanoblitz3D. This method enables generating high-resolution spatial H and E maps over large areas (up to hundreds of microns) by performing up to thousands of indents. Each indent test took up to 1 second, including the surface approaching, loading, unloading, retracting, and positioning the sample for the next test. A diamond Berkovich tip (Synton-MDP, Switzerland) was used for the nanoindentation tests. Several trial-and-error nanoindentation experiments were conducted to identify an optimal indentation depth and indent spacing. In our study, we performed the indentation mapping to capture the intrinsic H and E of the α and β phases in a Ti-6Al-2Zr-V-Mo alloy. The ratio of indent spacing to indentation depth is larger than 10 to avoid any overlapping effect between indentations. A maximum load of 1 mN, which results in a penetration depth ranging between 50 and 100 nm for α and β grains, and 500 nm spacing between the indents were chosen for all the tests. The H and E of α and β phases in the as-deposited and heat-treated samples are obtained from the same load, which are valid for comparison. The nanoindentation mapping with ~10,000 indents (50×50 μm^2 areas) and ~20,000 indents (50×100 μm^2 area) were carried out on the as-deposited and heat-treated samples, respectively. The indented areas of both samples contain more than 10 α and β grains to obtain microstructure-property correlations. The hardness (H) and reduced elastic modulus (E_r) at each test location can be calculated using the standard Oliver and Pharr method. The E_r includes the elastic contributions of both the indenter and the indented materials.

Keywords: Additive manufacturing · Titanium alloys · Nanomechanics

1 High-Speed Nanoindentation Mapping

High-speed nanoindentation mapping was carried out using a nanoindenter (iMicro, KLA Tencor, USA) using the method of Nanoblitz3D. This method enables generating high-resolution spatial H and E maps over large areas (up to hundreds of microns) by performing up to thousands of indents. Each indent test took up to 1 second, including the surface approaching, loading, unloading, retracting, and positioning the sample for the next test. A diamond Berkovich tip (Synton-MDP, Switzerland) was used for the

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nanoindentation tests. Several trial-and-error nanoindentation experiments were conducted to identify an optimal indentation depth and indent spacing. In our study, the indentation depth should be more than 50 nm to capture the intrinsic H and E of the α and β phases, while the ratio of indent spacing to indentation depth should be larger than 10 to avoid any overlapping effect between indentations [1]. A maximum load of 1 mN, which results in a penetration depth ranging between 50 and 100 nm for α and β grains, and 500 nm spacing between the indents were chosen for all the tests. The H and E of α and β phases in the as-deposited and heat-treated samples are obtained from the same load, which are valid for comparison. The nanoindentation mapping with ~10,000 indents (50 × 50 μm^2 areas) and ~20,000 indents (50 × 100 μm^2 area) were carried out on the as-deposited and heat-treated Ti-6Al-2Zr-V-Mo (TA15) samples [2], respectively. The indented areas of both samples contain more than 10 α and β grains to obtain microstructure-property correlations. The selected microstructures and resulted micromechanical properties of both samples are representative. The hardness (H) and reduced elastic modulus (E_r) at each test location can be calculated using the standard Oliver and Pharr method [3]. The E_r includes the elastic contributions of both the indenter and the indented materials, as expressed by the following equation:

$$\frac{1}{E_r} = \frac{\left(1-v^2\right)}{E} + \frac{\left(1-v'^2\right)}{E'} \tag{1}$$

where E' (1141 GPa) and v' (0.07) are the elastic modulus and Poisson's ratio of the diamond Berkovich indenter, respectively. The Poisson's ratio v of the TA15 alloy is ~0.39 [2]. Because the calculated E is about 98% of the E_r in this work, the E_r is used in this work to approximately represent the E of the TA15 alloys.

2 Microstructure Characterization

The microstructures and nanoindentation impressions of both as-deposited and heat-treated samples were imaged using a scanning electron microscope (SEM, Hitachi 3500, Japan) equipped with a backscattered electron (BSE) detector. Phases in the as-deposited and heat-treated samples were determined by X-ray diffraction (XRD, Rigaku MiniFlex 600) over a 2θ range between 30° and 90° at a scanning rate of 0.025° s^{-1}. The quantitative chemical compositions of α and β phases and the element maps were acquired using an electron probe microanalyzer (EMPA, JEOL JXA8230).

3 Results and Discussion

Figure 1a shows a schematic of the Laser Melting Deposition (LMD) process, and Fig. 1b, c present the high-speed nanoindentation mapping process and nanoindentation impressions, respectively. Prior to the nanoindentation tests, we characterized the

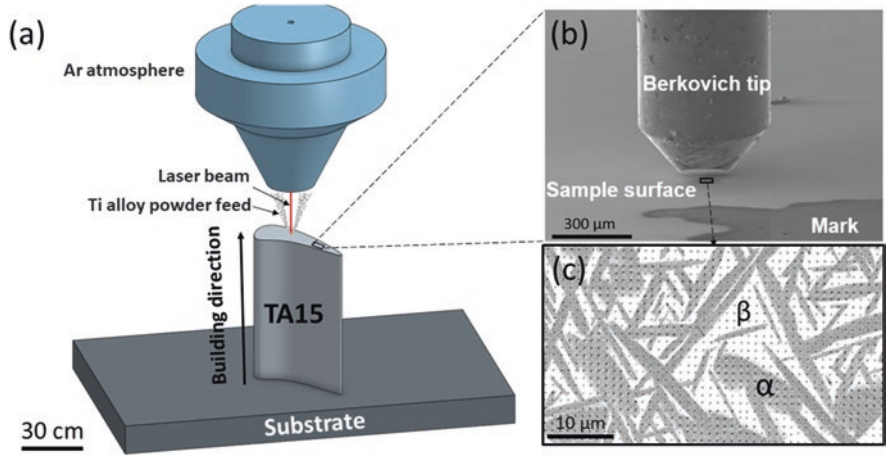

Fig. 1. (**a**) A schematic of the LMD system for additive manufacturing of the titanium alloy (TA15). (**b**) The SEM image of the nanoindenter with the Berkovich tip on the titanium sample surface. (**c**) The BSE image revealing the nanoindentation impressions on the α and β phases of the TA15 sample after the high-speed nanoindentation mapping.

pre-indent regions using Electron Probe Microanalyzer (EPMA) to elucidate the contribution of chemical composition to the resulting H and E values of α and β grains.

Figure 2 shows the microstructure, the corresponding H and E maps of the as-deposited TA15 sample [4]. The majority of the α grains, far larger than the β grains, can be captured and correlated with the spatial H and E maps (Figs 2a, b and d). For example, the three α grains, intersected by the line AB in Fig. 2a, involve tens of indents, showing the profile in the H and E maps. We can accurately extract the H and E values along with the line AB by point-to-point analysis, as shown in Fig. 2c. The α grains show distinct H and E values. High-resolution H and E maps also virtualize the morphology of the α and β grains, including a clear profile of the α/β boundaries. It is believed α/β boundaries are critical to determine the local deformation mechanism. Here, the high-speed nanoindentation mapping enables to measure the H and E distribution in the vicinity of the α/β boundary. It should be noted the effect of the α/β boundary on the H and E characterization is neglectable according to the absence of H or E change when approaching α/β boundaries (Fig. 2). Figure 3 presents the microstructure, corresponding H and E maps, and Al content of the heat-treated TA15 sample. The excellent correlation between the microstructure and mechanical property maps (H and E) is attributed to the large size of α and β grains. The contrasting H and E in the $\alpha+\beta$ microstructure are clearly observed. The α grains exhibit distinct H and E, which is consistent with the observation in the as-deposited sample. The homogeneous Al distribution in the α grains excludes the contribution of the solid solution strengthening to the contrasting H and E of the α grains. The H and E contrasts in the α grains should stem from the anisotropy of the hcp crystal structure. In addition to the titanium alloy, high speed nanoindentation mapping tests can be also used to capture grain-sclae mechanical properties in other multiple phase alloys, *e.g.,* graded γ-TiAl/Ti2AlNb alloys [5] and steel [6].

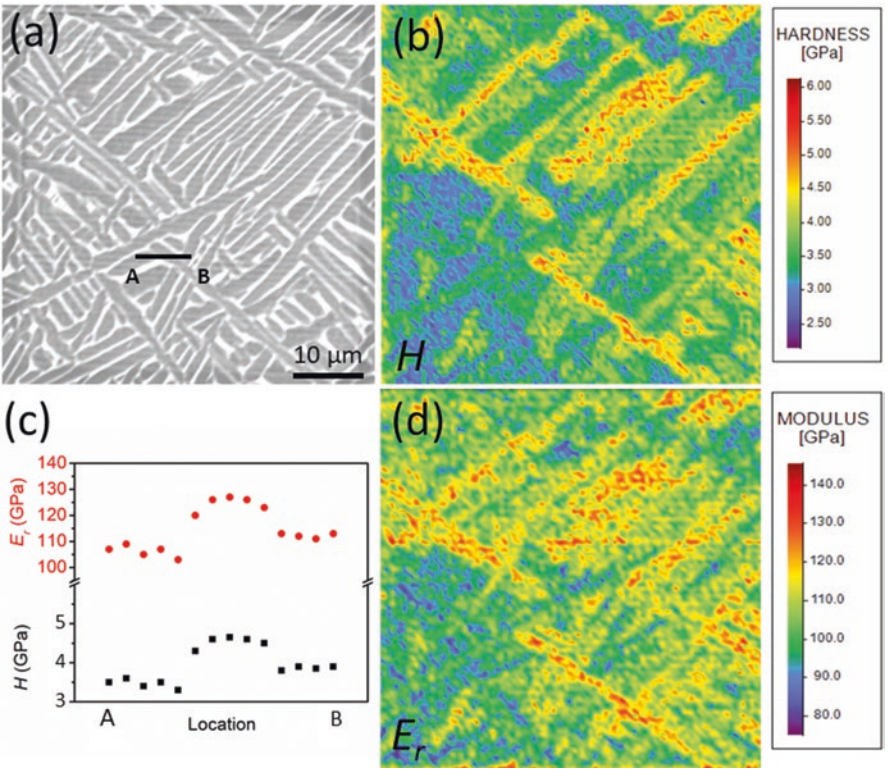

Fig.2. (**a**) BSE image of the as-deposited TA15 alloy. (**b**) The hardness (H) and (**d**) elastic modulus (E) of the rectangle region in (a). (**c**) The H and E values along with the line AB in 3(a), which involves three α grains.

4 Conclusions

In this study, we employ the high-speed nanoindentation technique to characterize the local mechanical properties (hardness and Young's modulus) of a near-α titanium alloy, Ti-6Al-2Zr-Mo-V or TA15, made by laser melting deposition. The following conclusions are drawn:

1. The high-speed nanoindentation mapping is an effective approach to reveal mechanical contrasts (*H* and *E*) of multiphase alloys with complex microstructures, such as 3D printed titanium alloys. The speed of indentation can reach approximately 1 second per indent (10,000 indents for ~2.5 hours). The special resolution can reach the submicrometer regime (~300–500 nm). For the heat-treated TA15 sample, this method provides the *H* and *E* distributions, clearly correlates the dual-phase ($\alpha + \beta$) microstructure, reveals the anisotropy of the hcp α phase. This method shows a limited mechanical-phase correlation for the as-deposited sample due to its ultrafine microstructure (grain size ~ 1 μm).

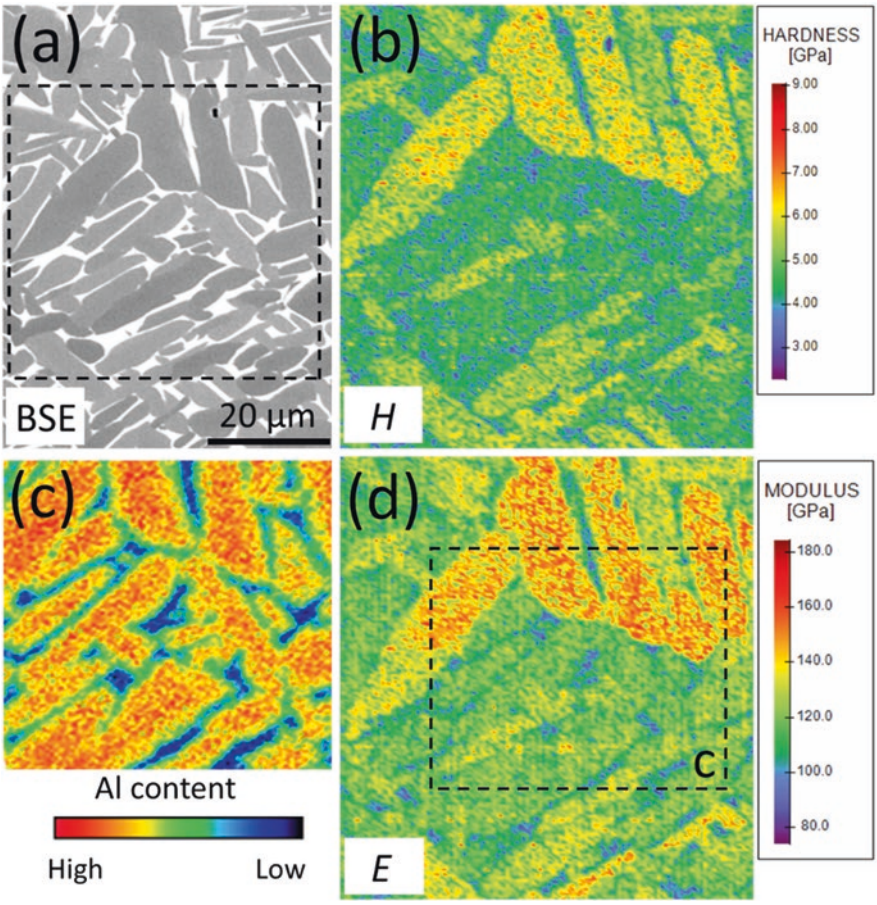

Fig. 3. (**a**) BSE image of the heat-treated TA15 alloy. (**b**) The *H* and (**d**) *E* maps of the rectangle region in 3(a). (**c**) The distribution of Al content in the rectangle region in 3(d), as measured by EPMA.

2. Using such a method, we observe that heat treatment leads to a concurrent hardening of the α and β phases**,** increasing by 32% and 12%, respectively. This phenomenon is associated with the redistribution of alloying elements: Al diffuses from the β phase into neighboring α phase, while Mo and V diffused along the opposite direction. The element redistribution also results in the increment of the *E*, by 4% in the β phase and 14% in the α phase. This mechanical distribution cannot be easily revealed using conventional indentation methods.
3. Toward broad applications, the high-speed nanoindentation technique, together with advanced microstructure characterization and machine learning tools, brings unique opportunities to study and design new multiphase materials across length scales.

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