

## AMBIENT-TEMPERATURE INDENTATION CREEP OF AN ADDITIVELY MANUFACTURED Ti-6Al-4V ALLOY

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### Abstract

Instrumented indentation testing technique is a robust, convenient, and non-destructive characterization method to study time-dependent plastic deformation in metals and alloys at ambient and elevated temperatures. In this current research, the depth-sensing indentation creep behavior of additively manufactured Ti-6Al-4V alloy and its mechanism were studied at ambient temperature for different additive manufacturing scan direction and scan size. Indentation creep tests were conducted through a dual-stage scheme (loading followed by a constant load-holding) at different peak load of 250, 350, and 450 mN with holding time of 400 s. In addition, microstructural quantitative analyses, using optical microscopy and scanning electron microscopy, were performed. Microstructural assessments and depth-sensing creep characterizations were then used to assess processing parameter/ microstructure/ creep properties relationships for this alloy.

**Keywords:** Instrumented indentation, Additive manufacturing, Ti-6Al-4V, Creep stress exponent.

### 1. Introduction

Titanium (Ti) has been a metal of interest for transportation applications and biomedical industries for its excellent physical and mechanical properties, and biocompatibility [1], [2]. Ti-6Al-4V alloy, out of different Ti alloy, contributes for more than half of all commercial Ti applications [1]. Additive manufacturing (AM), a layer-wise material manufacturing method, seems to be a suitable replacement for the conventional manufacturing processes of the Ti-6Al-4V alloy since conventional manufacturing processes are quite challenging. AM process differing from conventional manufacturing processes in terms of cooling rate during and thermal processing required assessing the relationship between the microstructure, processing parameters, and properties of the additively manufactured Ti-6Al-4V alloys to determine its ability to meet demands of engineering design considerations.

Consistency of mechanical properties and microstructures in additive manufacturing is still a critical issue and for two-phase Ti-6Al-4V alloy mechanical properties can be significantly varied by tailored microstructure [3]. The instrumented indentation testing technique (i.e. micro/nano-indentation) is considered a robust, convenient, and non-destructive testing method to

examine the microstructure/ mechanical property correlation in metals at ambient (room) and elevated temperatures including time-dependent plastic deformation. Nano/ micro-mechanical properties could be calculated from the load (P) and displacement (h) data recorded throughout the process in the form of load vs displacement curve [4], [5].

In this study, creep parameters including indentation creep behavior, creep rate, creep stress exponent ( $n$ ) have been analyzed at different AM scan directions and scan sizes by using depth-sensing (instrumented) indentation technique, a reliable, convenient, and non-destructive testing technique to examine the microstructure/ mechanical property correlation in metals at ambient (room) and elevated temperatures [4].

## 2. Experimental procedures

The material studied in this research is an AM Ti-6Al-4V alloy fabricated via the laser powder bed fusion (L-PBF) process, a well-developed AM technique [6], in both horizontal and vertical scan direction. Two rectangular ‘Big sample’ and two rectangular ‘Small sample’ were prepared for both X (horizontal) and Y (vertical) scan direction. Samples with tracks printed parallel to flow (argon) direction were named as “Core No Post X-big” (CNPX-big) and “Core No Post X-small” (CNPX-small) and tracks printed perpendicular to flow direction were named as “Core No Post Y-big” (CNPY-big) and “Core No Post Y-small” (CNPY-small). “No post” contour exposure was done on these specimens. Prior to the indentation, the surfaces of the specimens were scratch free mirror-like surface finish resulting from carefully ground with a series of progressively finer sand papers followed by fine polishing.

In this study, at ambient temperature (298 K) using a U9820A Keysight Nano-Indenter G200, instrumented indentation creep tests with dual stage scheme (loading followed by constant load holding) were performed using a pyramidal Berkovich diamond indenter [4] of 65.3° face angle.

At a constant indentation loading rate of 10 mN/s, various peak loads of 250 mN, 350 mN, and 450 mN were applied with dwell (holding) time of 400 s as the creep time.

## 3. Results and discussions

### 3.1. Microstructure

AM Ti-6Al-4V is generally an  $\alpha$ - $\beta$  alloy characterized with prior  $\beta$  grains that produces in an epitaxial way through several layers, grain boundary  $\alpha$ , and  $\alpha$  lath size [7]. Fairly high cooling rates experienced by Ti-6Al-4V alloy manufactured by L-PBF processes generates an  $\alpha$ - $\beta$  lamellar structure associated with  $\alpha$ -phase lamellae in a  $\beta$ -phase matrix. Fig. 1 shows the optical microscopy images of one of the specimens studied in this paper (CNPY small).

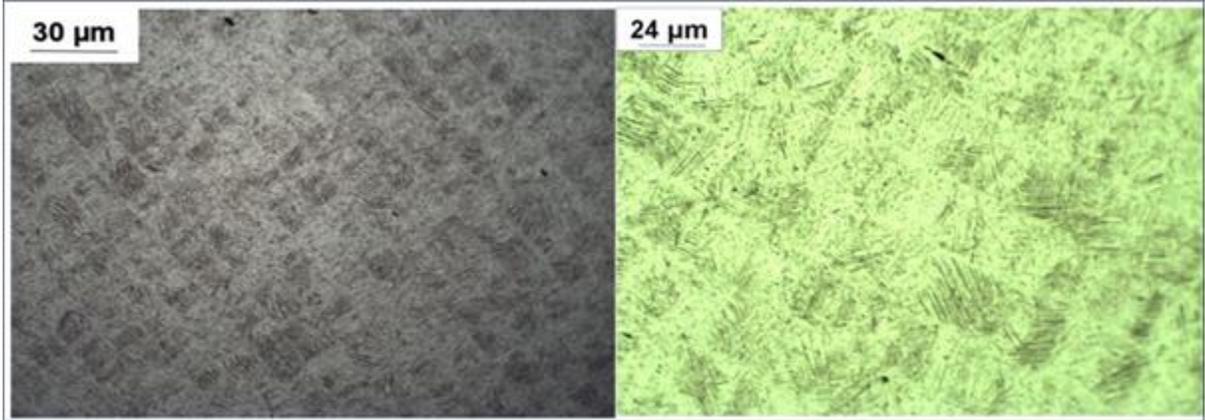


Fig. 1: Optical microscopy of microstructure of CNPY-small.

### 3.2. Indentation creep behavior

Indentation load/displacement at different peak loads of 250, 350, 450 mN along with the corresponding creep rate are shown in Fig. 2. It is worth mentioning that stress distribution in the depth-sensing indentation technique is much more complex than traditional creep tests, and at low displacement the maximum shear stress beneath the indenter exceeds the yield stress of the specimen (large tri-axial stresses in the range of some GPa) leading to creep in the materials even at ambient (room) temperature [8].

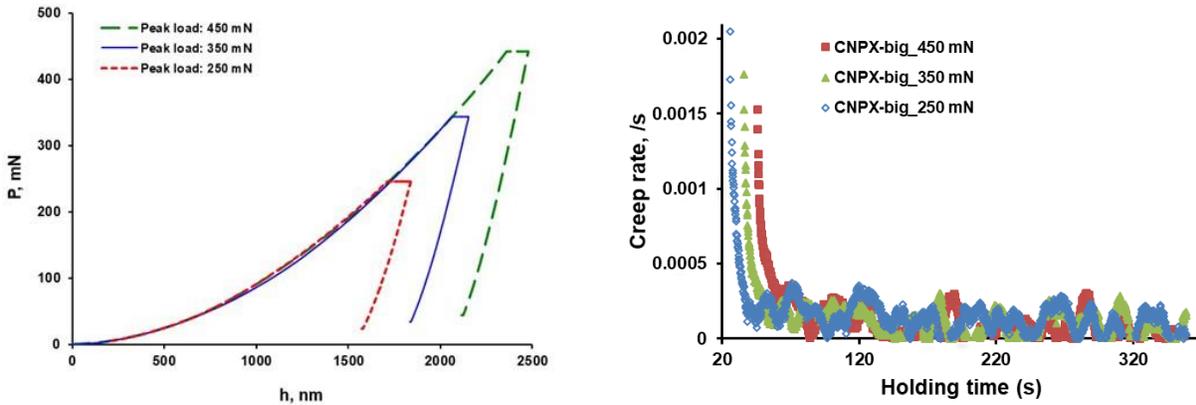
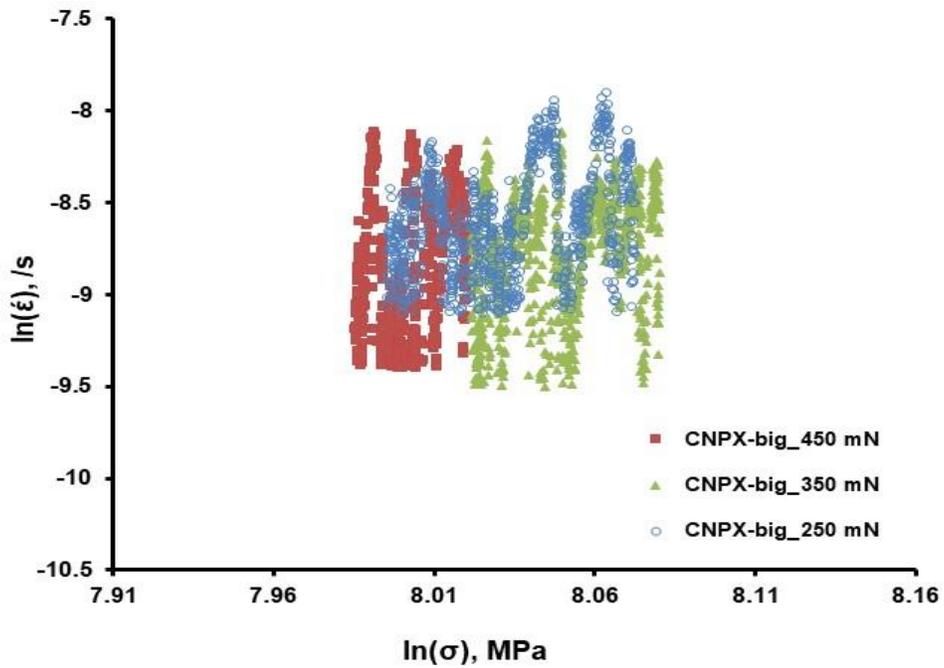


Fig. 2: Indentation load versus depth at different peak loads and the corresponding creep rate vs. dwell time for the CNPX-big sample. Transient and steady state-creep are observed in the creep rate data.

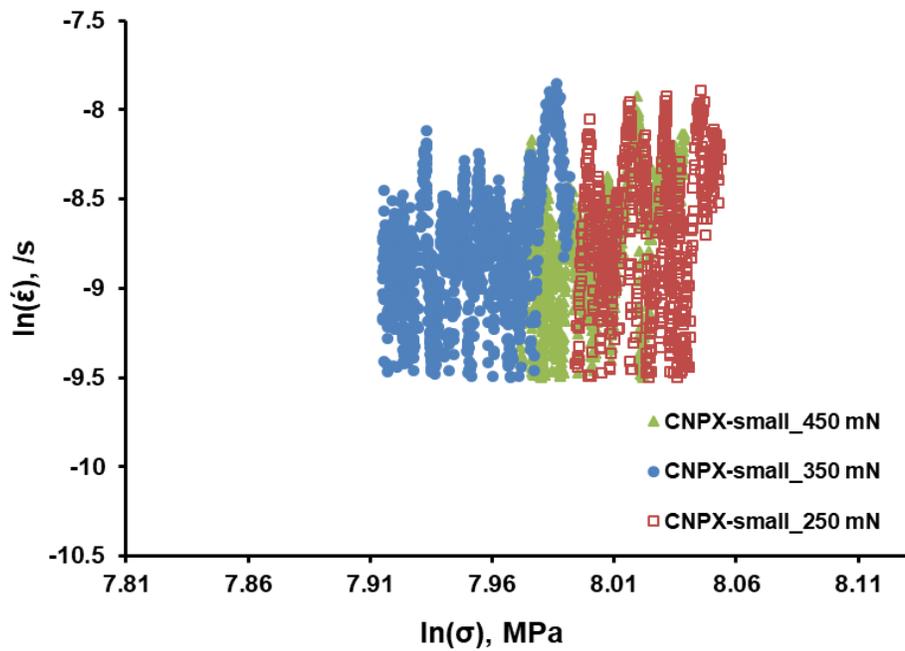
### 3.3. Creep stress exponent

Figures 3 a to d shows  $\ln \dot{\epsilon}_{ind}$  versus  $\ln \sigma_{ind}$  for all conditions studied in the present paper. Indeed, the creep stress exponent ( $n$ ) predicts the creep behavior illustrating creep stability and the dominant creep mechanism during depth-sensing indentation tests [9] which were deduced from the slope of  $\ln \dot{\epsilon}_{ind}$  versus  $\ln \sigma_{ind}$  curves in the steady-state creep region. Based on the calculated slopes, in all cases and conditions, the  $n$  values were recorded values greater than 3. This shows

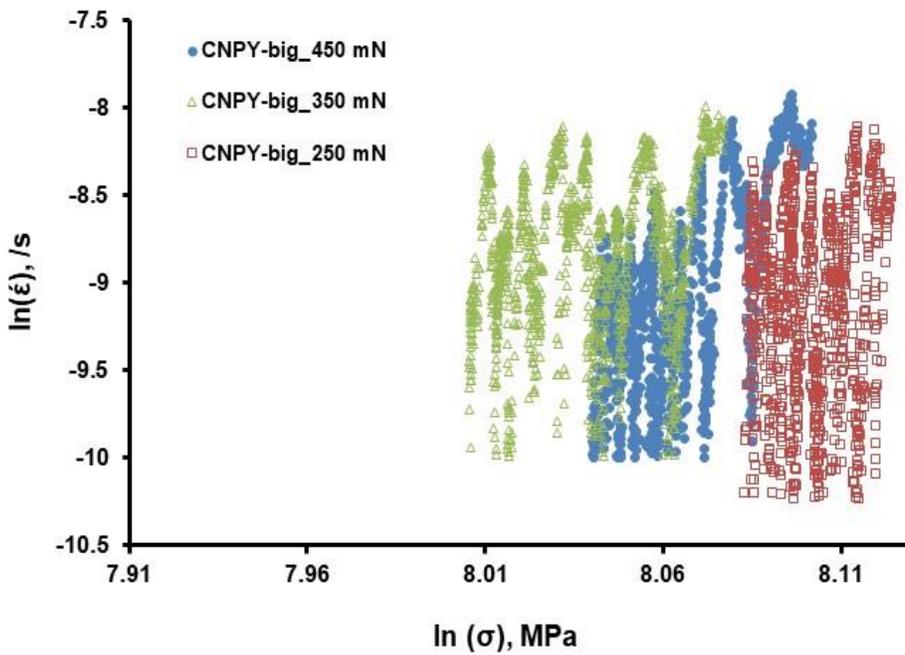
that the indentation creep mechanism for the printed Ti-6Al-4V materials at ambient (room) temperature is controlled through power-law (dislocation) creep [10]. Since the temperature here is well below the recrystallization temperature of Ti (*i.e.*  $50\%T_m$ ), the produced results are expected as no grain boundary sliding (GBS) and/or diffusion creep are involved here. It is worth mentioning that these data extracted from the steady state creep region and not necessarily the transient region.



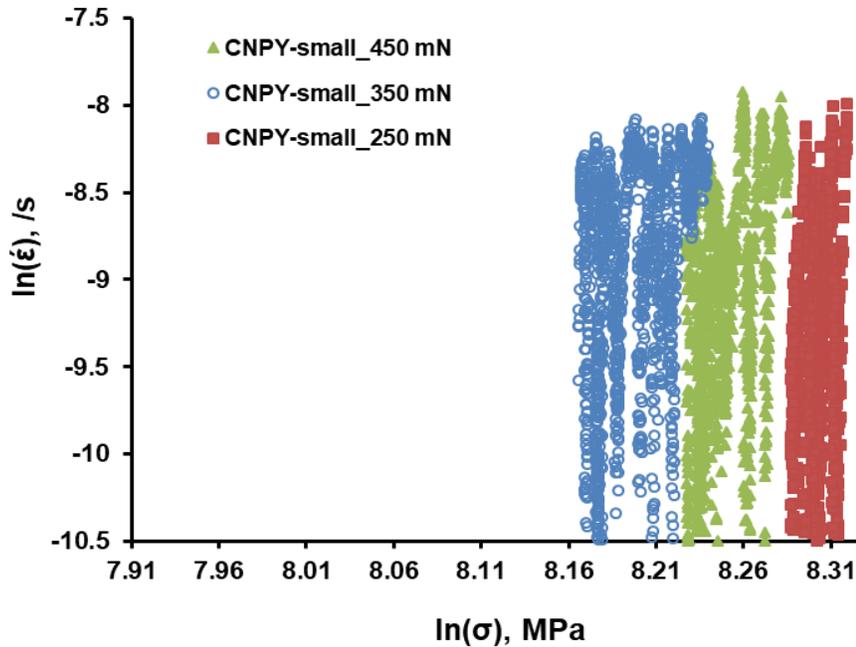
**a**



b



c



d

Fig. 3: Creep stress exponent (n) across different samples for (a) CNPX- big, (b) CNPX- small, (c) CNPY- big, (d) CNPY- small.

#### 4. Conclusions

The following conclusions were drawn from the of this investigation of AM Ti-6Al-4V alloy scanned with different print parameters using instrumented indentations:

- Creep parameters are function of indentation load and time during constant-load holding stage.
- At low displacement the maximum shear stress beneath the indenter exceeds the yield stress of the specimen
- Secondary stage of creep is dominated by dislocation movement (power-law creep).
- The creep stress exponent (n) increased as the indentation peak load increases.

#### Acknowledgement

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#### References

- [1] C. Leyens and M. Peters, *Titanium and Titanium Alloys*. 2003.
- [2] M. Niinomi, "Mechanical biocompatibilities of titanium alloys for biomedical applications," *Journal of the Mechanical Behavior of Biomedical Materials*, vol. 1, no. 1.

- pp. 30–42, 2008.
- [3] R. Filip, K. Kubiak, W. Ziaja, and J. Sieniawski, “The effect of microstructure on the mechanical properties of two-phase titanium alloys,” in *Journal of Materials Processing Technology*, 2003, vol. 133, no. 1–2, pp. 84–89.
  - [4] A. C. Fischer-Cripps, “Nanoindentation,” in *Nanoindentation*, 2011, pp. 21–38.
  - [5] C. A. Schuh, “Nanoindentation studies of materials,” *Mater. Today*, vol. 9, no. 5, pp. 32–40, 2006.
  - [6] S. Bremen, W. Meiners, and A. Diatlov, “Selective Laser Melting. A manufacturing technology for the future?,” *Laser Tech. J.*, vol. 9, pp. 33–38, 2012.
  - [7] F. Wang, S. Williams, P. Colegrove, and A. A. Antonysamy, “Microstructure and mechanical properties of wire and arc additive manufactured Ti-6Al-4V,” *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.*, vol. 44, no. 2, pp. 968–977, 2013.
  - [8] W. B. Li, J. L. Henshall, R. M. Hooper, and K. E. Easterling, “The mechanisms of indentation creep,” *Acta Metall. Mater.*, vol. 39, no. 12, pp. 3099–3110, 1991.
  - [9] R. Schwaiger, B. Moser, M. Dao, N. Chollacoop, and S. Suresh, “Some critical experiments on the strain-rate sensitivity of nanocrystalline nickel,” *Acta Mater.*, vol. 51, no. 17, pp. 5159–5172, 2003.
  - [10] M. W. Barsoum, “Fundamentals Of Ceramics,” *Vasa*, p. 622, 2003.