

Letters

Surface property study of additively manufactured Inconel 625 at room temperature and 510 °C

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ABSTRACT

Various mechanical systems are subjected to inherent oscillatory motions often paired with extreme, high-temperature environments resulting in fretting wear. In this work, the fretting wear resistance of additively manufactured Inconel 625 was studied at room and elevated temperatures. In-situ temperature-controlled nanoindentation was employed to further elucidate mechanical property evolution as a function of temperature. The results showed a 30% decrease in near-surface mean hardness at 510 °C compared to room temperature. However, the material exhibited significantly lower wear and coefficient of friction values at high temperatures, attributed to the formation of a compacted, intermediate oxide layer preventing excessive friction and wear.

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1. Introduction

Nickel-based superalloys with superior thermochemical, mechanical, and tribological properties are of high importance for critical components in several applications such as gas turbine engines, propulsion shafts, welding products, and heat exchangers [1–4]. Such components are usually under inherent vibration leading to small amplitude displacements under normal pressure at interfaces which leads to a phenomenon called fretting wear [5]. Fretting wear is defined as wear arising due to the linear oscillating motion of relatively small amplitudes [6–8]. While wear and corrosion have been studied extensively for Inconel 625 coatings [9–13], there is a scarcity of available studies in the area of fretting/sliding wear of traditionally manufactured Inconel 625 components [14,15]. With additive manufacturing gaining traction in the modern manufacturing sector on a daily basis, interest has peaked among researchers to replace the conventional manufacturing process with additively-manufactured (AM) materials. There has been an extensive number of studies on discrete aspects of the AM Inconel 625 such as surface morphology [16–18], mechanical behavior [19–21], and microstructural evolution [22–24]. However,

limited investigations are available in tribological aspects [25,26] with no available study on contact behavior and fretting wear of AM Inconel 625 at high temperatures. Industrial applications of grade 1 Inconel 625 [27] such as in feedstock stock superheaters [28], flexible couplings in automotive exhaust systems [2], and many more applications generally operating between 400 °C and 650 °C are potential applications for future AM Inconel 625 adoption. This report attempts to achieve this goal by performing fretting wear tests at room temperature and 510 °C using a silicon nitride ball (as counterpart) on AM Inconel 625 paired with in-situ temperature-controlled indentation to study the evolution of surface mechanical properties correlating with temperature rise.

2. Experimental setup

The fretting tests were conducted using Bruker UMT-TriboLab consisted of an upper assembly with a carriage and pin holder and a lower assembly comprising of a drive and sample holder along with a temperature chamber as shown in Fig. 1(a). The carriage is mounted with a force sensor that can measure normal and lateral forces up to 200 N (Fig. 1(b)). The reciprocating drive was used to simulate a linear oscillatory motion capable of a stroke length of 0.1–25 mm with a frequency of up to 60 Hz. (details in [supplementary information \(SI\) section S1](#)).

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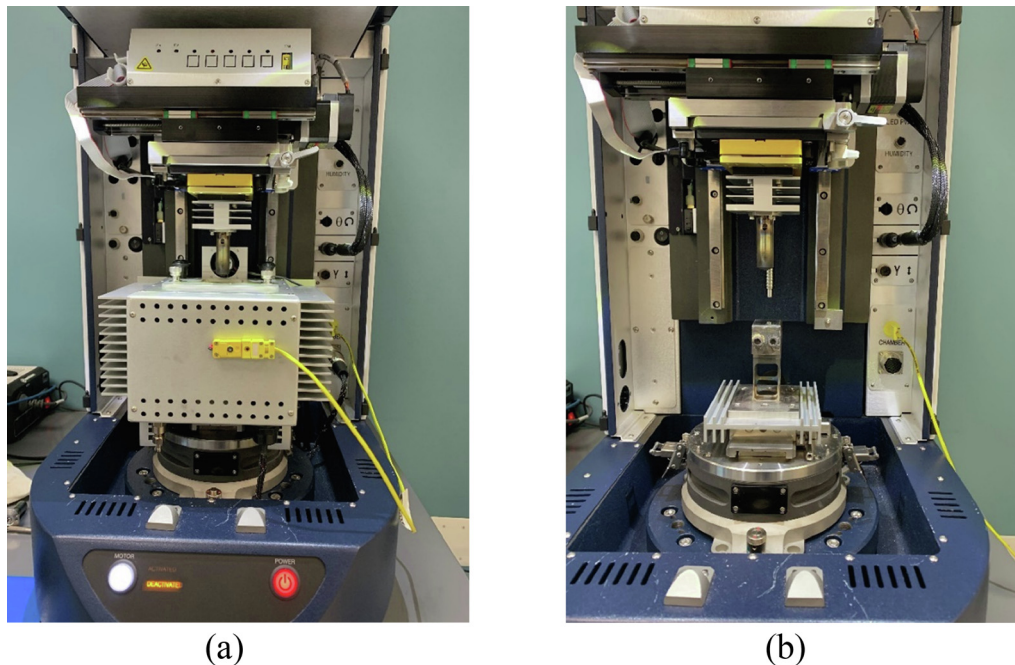


Fig. 1. Bruker UMT-TriboLab (a) with the temperature chamber, (b) without the temperature chamber.

3. Results and discussion

3.1. Fretting wear

Fig. 2(a) shows the representative in-situ coefficient of friction (CoF) with respect to time at room and 510 °C temperatures. The friction at room temperature begins at approximately 0.4 and fluctuates before it achieves steady-state at a value of 0.49. The fluctuations can be attributed to the initial running-in process with thin oxide layer removal as well as generation and accumulation of a high amount of debris in the contact area while surface asperities become truncated. This results in an increase in the contact area, leading to a relatively stable CoF. At an elevated temperature of 510 °C, fairly moderate fluctuations are observed, wherein the CoF begins at 0.35 and eventually reaches a steady-state value of 0.26. High consistency was observed between two test trials wherein an averaged steady-state CoF is found to be 0.48 at room temperature and 0.26 at 510 °C (Fig. 2(b)). At high temperature, CoF stabilizes in a shorter time (~500 s) as compared to the room temperature experiment (~1400 s). These CoF values were compared and found to be in line with the behavior of the wrought Inconel 625 investigated by Iwabuchi [14]. The low fluctuations, rapid stabilization, as well as lower CoF at elevated temperatures, can be mainly attributed to the formation of a glazed compacted oxide layer on the surface. The compacted oxide layer starts to form as the experiment begins where it becomes more uniform over time leading to a stable CoF. This stable, glazed layer acts as a lubricating protective intermediate layer separating direct contact ball-on-substrate thus reducing the friction between the contacting surfaces. The formation of this glazed layer has been observed for several nickel-based alloys including previous studies of the authors [14,29–35]. The SEM images (see Fig. S2 of SI) show the relatively uniform layer formed by the compaction of the wear debris trapped in the contact for the 510 °C track in contrast to distinct ploughing lines seen in the room temperature track. In addition, and to a lesser extent, stable and low CoF observation can be attributed to the increase in bulk material compliance at high temperatures (see Section 3.2) making the contact more conformal,

reducing local pressure, and thus decreasing resistant to the motion, and subsequently lowering deformation (ploughing) component of CoF that results in an overall smaller CoF.

Fig. 3(a) and (b) display representative wear scars for tests at room temperature and 510 °C, respectively. Fig. 2(c) and (d) show the maximum wear depth as well as wear volume for all the tests as well as the mean values. At room temperature, the maximum wear depth is higher than that of 510 °C. Similarly, the calculated wear volume at room temperature is about $14,970 \mu\text{m}^3$ where at 510 °C, it reduces to $6420 \mu\text{m}^3$. The observation of lower wear at elevated temperatures is similar to the trend found for CoF. This tribological behavior was also observed by Iwabuchi [14] for wrought Inconel 625 and also in other nickel-based alloys [29–32,36–38] including studies by the authors (Inconel 617 [34] and Incoloy 800H [35]). At 510 °C, the oxidation rate becomes considerable compared to room temperature due to the presence of sufficient air and an oxide layer forms on nickel-based alloys. This top layer mainly consists of nickel oxide and chromium oxide [33–35]. At high temperatures with sufficient contact pressure, oxide particles form a stable ‘compacted glazed oxide’ layer with good adhesion to the substrate which decreases the wear volume on the track [39] and acts as an intermediate protective layer between the silicon nitride ball and the substrate. This reduces direct contact with the substrate and mitigates the excessive amount of wear.

3.2. Nanoindentation

Fig. 4 displays both hardness and Young’s Modulus of the material at room temperature and 510 °C. Seven individual trials were conducted for each temperature regime wherein curve-fitting of the force–displacement curves was performed using the Oliver-Pharr method to extract relevant hardness and modulus information [40]. It can be seen that mean surface hardness and modulus decrease from 4.85 GPa to 3.49 GPa, and 203.50 GPa to 138.59 GPa as temperature increases from room temperature to 510 °C, which is in agreement with other reports elucidating the high-temperature deformation of Ni-based superalloys [41,42]. This

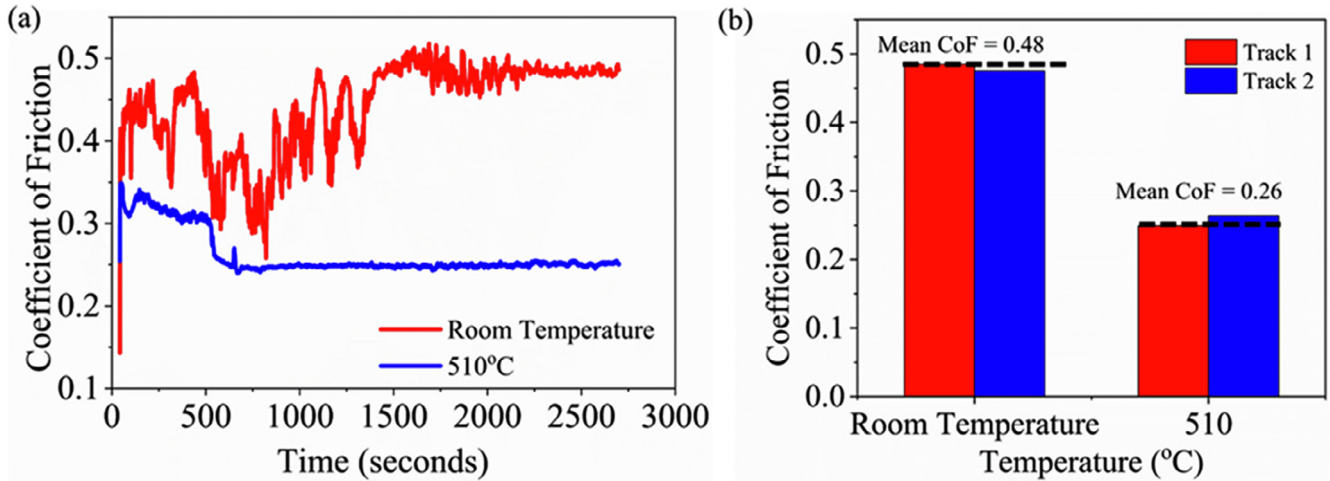


Fig. 2. (a) In-situ CoF and (b) average CoF for room and high-temperature conditions.

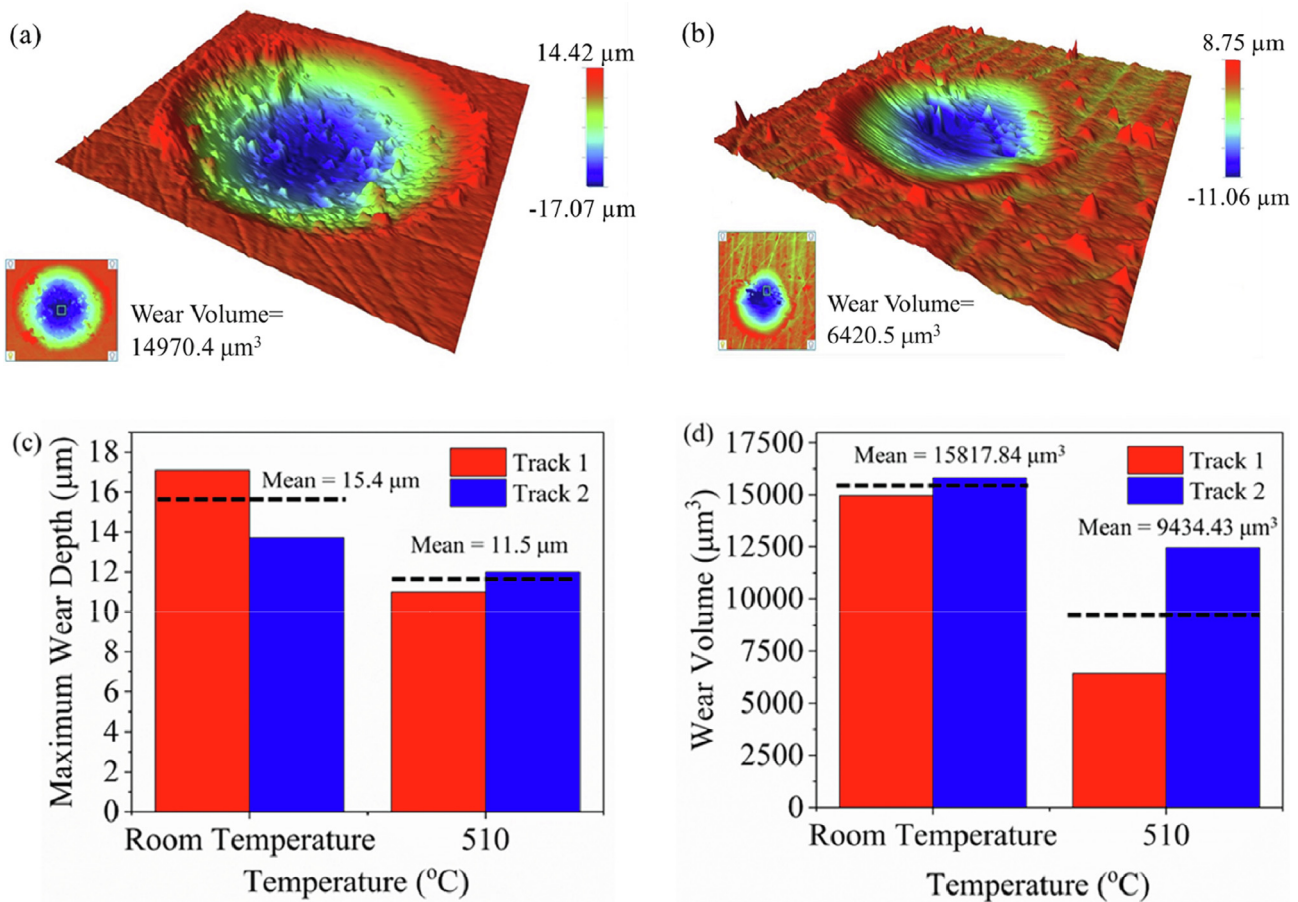


Fig. 3. Wear scars at (a) room temperature and (b) 510 °C; (c) maximum wear depth and (d) wear volume versus temperature.

observed reduction in both surface hardness and modulus is mainly attributed to the softening effect as a result of heat input. Specifically, in several variants of Inconel, it has been observed that moderate heat input in a short duration of time significantly overshadows the effect the strengthening phases, which ultimately increases ductility, making the material more susceptible to deformation [43]. It should be also added that 510 °C, is a low temperature for precipitation hardening to occur in Inconel 625 [44]. It

should be noted that this does not necessarily mean higher wear volume at elevated temperatures, as the oxide formulation is the most important factor to influence a tribo-pair behavior. Due to the formation of the compacted oxide layer, the increase in ductility does not influence the high-temperature wear behavior of AM Inconel 625 as characterized by the results presented in Figs. 2 and 3. Additional indentation results are illustrated in the Supplementary Information section S2.

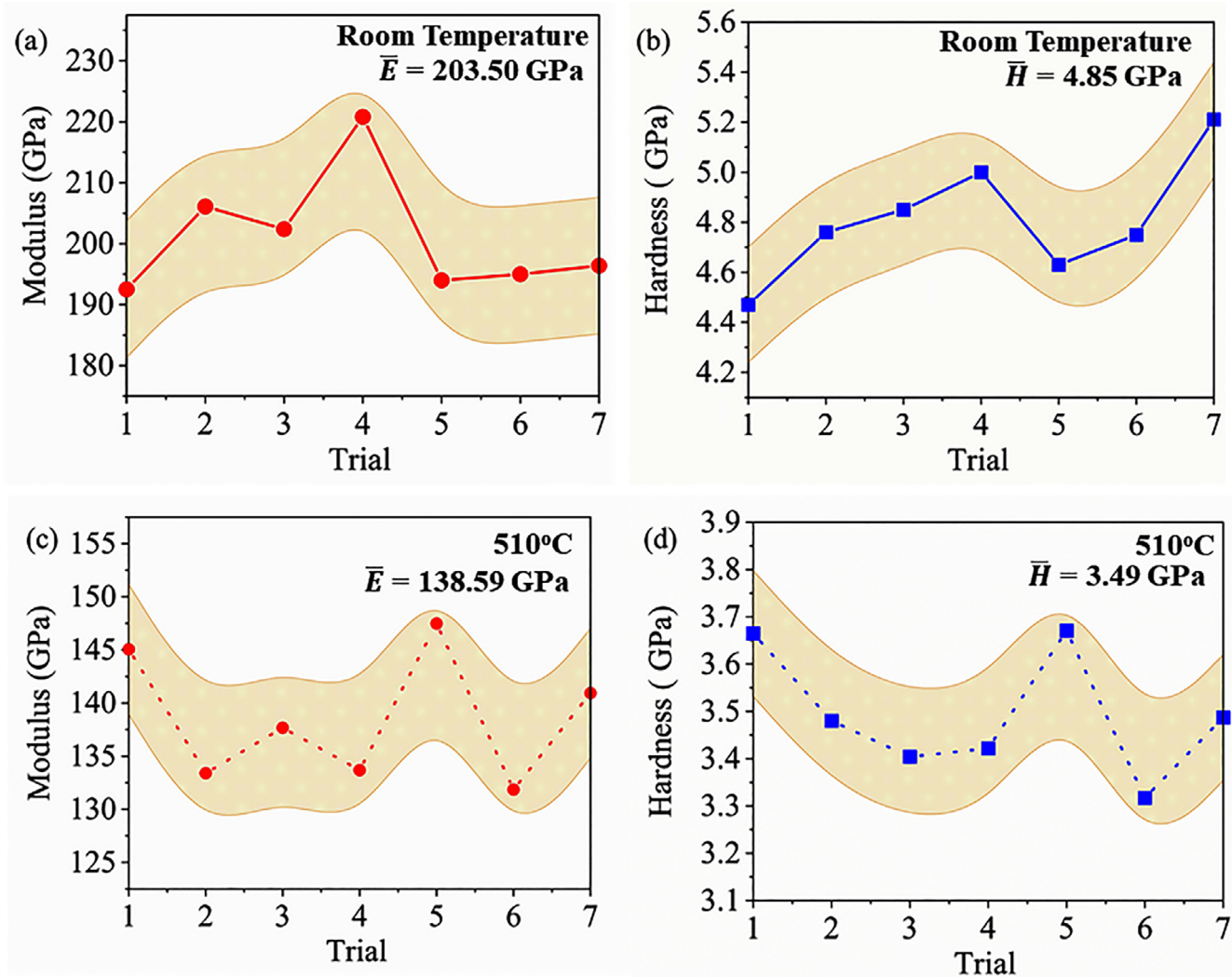


Fig. 4. Nanomechanical characterization of Inconel 625: (a) Young's Modulus and (b) hardness at room temperature, (c) Young's Modulus and (d) hardness at 510 °C. Error curves represent the standard deviation from each respective measurement.

4. Conclusion

Fretting wear tests coupled with in-situ temperature-controlled indentation were conducted on AM Inconel 625 at room temperature and 510 °C to establish its tribological and mechanical behaviors at elevated temperatures. While hardness and elastic modulus reduced significantly at 510 °C, the coefficient of friction and wear were found to be lower at 510 °C compared to room temperature tests. This is due to the formation of a compacted oxide layer acting as a lubricating stopgap layer minimizing direct contact between the ball and the substrate, reducing wear at elevated temperature. This report presents the first elevated temperature study on AM Inconel 625 and is a path forward to its effective incorporation in novel designs and complex components in many applications.

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.mfglet.2020.10.001>.

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