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RESEARCH

Effect of Cryogenic Treatment on the Wear Behavior of Additive Manufactured 316L Stainless Steel

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A B S T R A C T

In this research work, an investigation is done to study the effect of cryogenic treatment on 316L SS fabricated using Direct Metal Laser Sintering process. Cryogenic treatment is done at -196°C for 24 hours in a chamber filled with liquid nitrogen. The post effects of this cryogenic treatment over the properties of the material such as hardness, wear behaviour and microstructure have been studied. The result showed that the porosity present in additive manufactured 316L SS is greatly reduced after cryogenic treatment. Tribological properties were analyzed, using zirconia pin-on- additive manufactured 316L SS disc, the result showed that the treatment has considerable effect on decreasing coefficient of friction. Wear track surface morphologies shows the evidence for the indentation of the abrasives in pores in the untreated AM sample. Hardness increases significantly due to strain-induced phase change in the sample after cryogenic treatment.

1. INTRODUCTION

Additive Manufacturing (AM) is one of the advanced manufacturing processes and its use in many fields is taking a big leap [1]. AM process involves fabrication of final component only by adding up of material rather than involving cutting or removal of material from bulk material as generally seen in any conventional manufacturing processes [2]. AM process exhibits efficient use of raw material and produces minimal waste while reaching competitive cost of fabrication and time to manufacture [3]. In the AM process, design in the form of a 3D Computer Aided Design (CAD) model can be used directly for conversion of raw materials into a finished product without using any external fixtures and cutting tools [4]. AM holds out many advantages such as short cycle times and quick production rates over conventional manufacturing processes [5]. Applications of additive manufactured parts are known for tooling, jigs, and fixtures in the manufacturing industry to artificial biological human body implants in the medical industry [6, 7]. Direct Metal Laser Sintering (DMLS) is a powder bed fusion based AM process in which an object gets built up from its powder material due to the sintering of the powder material in a layer by layer fashion [8]. The Laser sinter the selected areas in the metal powder spread on the powder bed to become a section of the final built and then followed by spread of a new layer of powder.

Cryogenic treatment is basically the process of treating work pieces at an extremely low temperature range such as -180°C to -196°C using liquid nitrogen in order to improve wear resistance, corrosion resistance and dimensional stability [9]. Cryogenic treatment has applications in various fields due to its cryogenic hardening mechanism [10]. Mechanical properties such as hardness and wear properties have been observed improvement to post cryogenic treatment in the case of materials processed through conventional manufacturing processes, i.e. casting, forging and rolling operations. The advantages of cryogenic treatment include longer part life, reduced failure because of cracking, increases in corrosion resistance, and decreases in the coefficient of friction, reduced creep and improved machinability [11-14]. It has applications in cutting tools, forming tools, surgical tool and music industry [15, 16].

Additive manufacturing involves a different principle of building up parts (layer by layer addition of material), which is opposed to that of the conventional process where material is removed, cast or forged. This built up strategy may have a difference in between the performance of additive manufactured parts and conventional manufactured parts. In AM 316L SS, volumetric percentage of porosity is significant and distinct compared to conventional processed material [17,18]. Techniques such as hot isostatic pressing in post processing [19] and grain-boundary strengthening using reinforcement in metal powder [20] was used to improve the performance of AM parts. On the other hand, cryogenic treatment of conventional processed 316L SS has improved performance due to precipitation of carbides along the grain boundaries and transformation of γ phase of Face centered cubic austenite to α' phase of body centered tetragonal ferrite or to a ε phase hexagonal closed pack martensite [21, 22]. However, effect of cryogenic treatment over the additive manufactured metals does not have any elaborate exploration, despite of profound study that has been seen on the effect of cryogenic treatment over conventionally manufactured 316L SS. So, in this work, additive manufactured metal part is considered for cryogenic treatment, and then its effect on wear behavior of properties such as hardness, microstructure and wear resistance is investigated.

2. EQUIPMENTS AND TECHNIQUES

2.1 Method & Material

In DMLS process layer by layer sintering of metal powder takes place by using thermal energy of the laser with the power of 200-400 W. This thermal energy of subsequent laser scans is just sufficient to join a portion of the previously sintered region, which ensures well bonded, high density structures from engineering metals [23,24]. DMLS - EOSINT M280 machine is used for part fabrication. Part fabrication through the DMLS process requires the CAD model of the part in STL file format as an input. Hence the model as shown in Fig. 1 is done using the CAD modeling software.

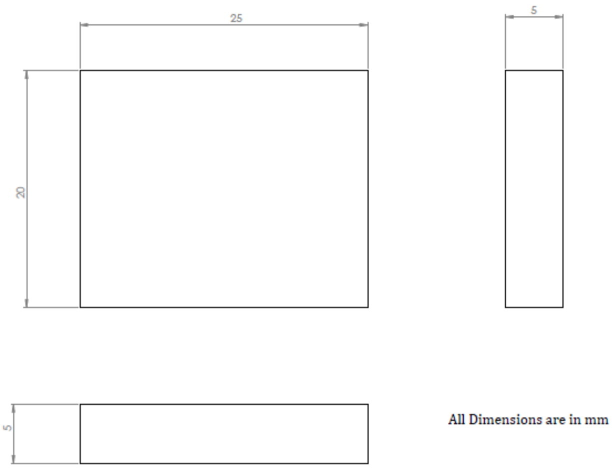


Fig. 1. Isometric view of CAD model for specimen fabrication in DMLS.

Table 1. Chemical composition of additive manufactured 316L SS parts.

SL.No.	Elements	Weight %
1	Cr	19
2	Ni	15
3	Mo	3
4	C	0.030
5	Mn	2.00
6	Cu	0.50
7	P	0.025
8	S	0.010
9	Si	0.75
10	N	0.10
11	Fe	Balance

The material used in this study is 316L stainless steel (SS) which is known for its usage in a wide array of industries due to its corrosion resistance, biocompatibility, malleability and heat resistance at less cost [25]. 316L SS is used mainly in critical applications such as orthodontics pins & screws, orthopedic implants like total hip and knee replacements and as a fin in the cryogenic loop heat pipe for space application [26-28]. The advantages of the DMLS process to produce parts with complicated spatial forms of powder materials further enhances the applications of 316L SS [29]. The chemical composition of the AM 316L SS part is summarized in Table 1. For this study, 316L SS specimen as per the CAD model has been manufactured using the DMLS process.

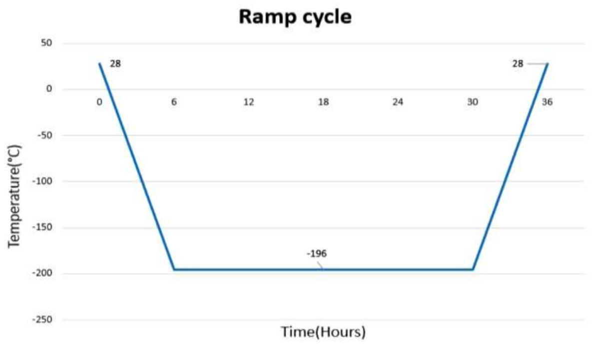


Fig. 2. Ramp cycle followed for cryogenic treatment of DMLS part.

The rest of the study is based on the effect of the cryogenic treatment over the surface properties of the additive manufactured 316L SS. So, the part fabricated using the DMLS process is taken for cryogenic treatment which includes a basic cycle which is known as the Ramp cycle. It involves three major steps; ramp down, soak and the ramp up. In this case, the ramp cycle parameters are as shown in Fig. 2. The part was initially ramped down, i.e. its temperature was dropped down. The cooling down was done using nitrogen vapor slowly from the ambient temperature (28 °C) to the cryogenic temperature (-196 °C). The duration of the ramped down cycle was 6 hours, i.e. gradual rate of drop in temperature is 0.62 °C/min. The next step was the soak. Here the component was soaked in a chamber filled with liquid nitrogen. The sample was soaked for about 24 hours as indicated in existing literature [30,31]. In this stage, the temperature was kept constant at around -196°C. The final step was the ramp up. Here the component was taken up from -196 °C to ambient temperature of 28 °C. During the ramp up step, the rate of increase in temperature was 0.62 °C/min.

2.2 Micro hardness test

Vickers hardness was measured for both the cryogenic treated as well as the non-treated samples to understand the effect of cryogenic treatment over additive manufactured 316L SS. Parameters for the Vickers test included a dwell of 10s, while the load was 0.5kg. Micro hardness was measured at various points on the surface observed through the microscope.

2.3 Reciprocating wear test

Cryogenic treatment reveals to have a positive impact over wear resistance and wear properties of materials. The part was taken for wear test, following cryogenic treatment. The wear test was done through a computer assisted reciprocating wear tester machine. The machine used was DUCOM TR-285-M1. Tests were carried out according to the ASTM G 133-05, Standard Test Method for Linearly Reciprocating Ball-on-Flat Sliding Wear. The wear test was conducted for 30 minutes on cryogenic treated and non-cryogenic treated parts. AM 316L SS slab of 30 mm X 20 mm X 10mm was used against the zirconia ball with 6 mm diameter. The wear tests were carried out in air at ambient temperature and dry conditions with a constant load of 5 N. The frequency of reciprocating motion was set at 2 Hz and the sliding distance was set to 15 mm giving a sliding velocity of 40 mm/s. The friction coefficient was evaluated using 'Winducom 2006' machine associated software.

2.4 Microstructure Study

Microstructure analysis of both the cryogenic treated and untreated AM sample was done to understand the microscopic changes in additive manufactured material after the cryogenic treatment. Oxalic acid was used as an etchant for observing microstructure. It was prepared using 10 g of oxalic acid powder mixed with 100 ml of distilled water. Further, microstructure analysis study involved a combined study of SEM (Scanning Electron Microscope) analysis and EDAX (Energy Dispersive Analysis of X-Rays). SEM and EDAX were taken at various locations in order to understand the surface morphology and the composition of the wear track of DMLS sample. To understand the phase change in material after the cryogenic treatment, surface residual stresses were measured by using X-ray diffraction (XRD) system (PULSTEC/μ-*X360*).

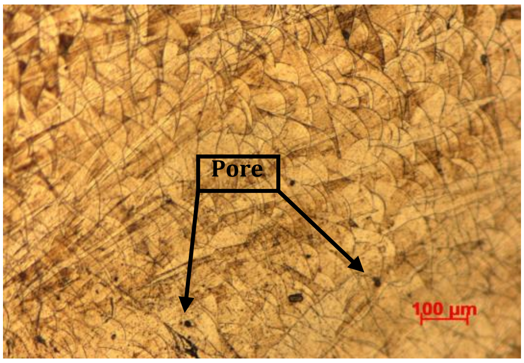
2.5 Surface roughness test

Analysis of surface roughness of the wear track was done using Taylor-Hobson non-contact surface roughness tester for getting an understanding of the phenomenon behind the coefficient of friction and wear rate. Magnification of 10X was used to study the surface roughness parameters within a 0.08 mm^2 area of wear track of additive manufactured 316L SS sample before and after cryogenic treatment.

3. RESULTS AND DISCUSSIONS

3.1 Microstructure

Microstructure of conventional manufactured [32] and additive manufactured 316L SS specimen is shown in Fig. 3a & 3b. Distinct grain boundary as observed in conventional manufactured 316L SS was not observed in Additive Manufactured 316L SS. In contrast, AM specimen displayed repeated crescent shape. This observance was due to the exposure of the laser in a powder bed that takes Gaussian boundary profile as shown in Fig. 3c. A Similar phenomenon was reported in the literature available on the microstructure study of AM components [33].



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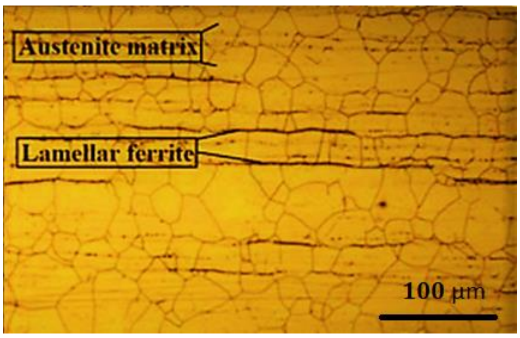

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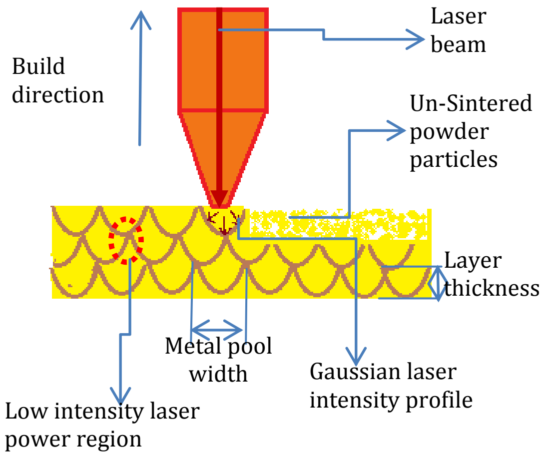


c)

Fig. 3. a) Additive manufactured 316L SS microstructure and b) conventional manufactured 316L SS microstructure [32] & c) schematic representation of sintering mechanism in DMLS process.

The microstructure clearly displays the presence of sinter pools. Sinter pools are the repeating pattern observed due to the successive sintering of powders within a layer as well as layer by layer. In addition, pores are majorly observed at the intersection of Gaussian boundaries where the laser penetration is getting reduced due to Gaussian profile.

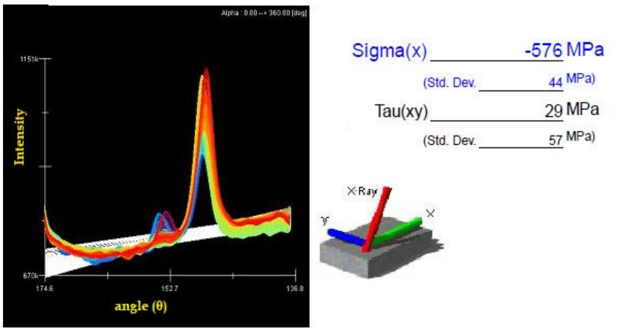
3.2 Micro hardness

Vickers hardness was noted at different locations for each of the two cases, namely cryogenic treated and non-treated sample. Intender impression of the samples is shown Fig. 4. The results of the micro hardness in terms of Vickers hardness (HV) for both the untreated and cryogenic treated are summarized in Table 2.

Fig. 4. Micro Vickers's diamond intender indentation on Additive manufactured SS 316 L specimen.

Table 2. Micro Vickers's hardness of additive manufactured 316L SS before and after cryogenic treatment.

Properties	Before Cryogenic treatment	After Cryogenic treatment
Vickers's Hardness (HV)	242.84	312.15



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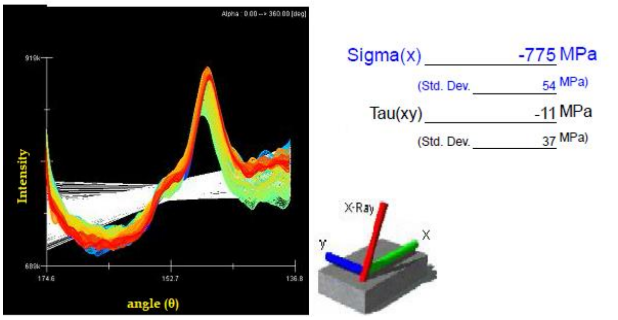


b)

Fig. 5. X-ray diffractometer (XRD) pattern and residual stress of the AM 316L SS samples a) before cryogenic treatment and b) after cryogenic treatment.

In the post cryogenic treatment the hardness for the additive manufactured 316L SS component was seen to increase by 28.5 %. As observed in conventional manufacturing process, the cryogenic treatment on the additive manufactured sample, displayed a significant improvement in hardness. Fig. 5 represents the X-ray diffractometer (XRD) pattern of the AM 316L SS samples before and after cryogenic treatment. From the pattern, it is observed that peak intensity is reduced and the diffraction angle broadening for cryogenic treated sample. Change in the pattern indicating the severe plastic deformation (SPD) in AM sample due to the formation of strain-induced martensite in the austenite matrix after cryogenic treatment [34]. In addition, residual stress is measured in both samples to observe the strain-induced shrinkage in terms of the quantitative result. It shows that 34.54 % increases in compressive residual stress of the cryogenic treated sample. This could be the phenomenon behind the increase in hardness of cryogenic treated AM 316L SS sample. The additive manufactured specimen is further investigated through SEM analysis to understand the other changes after cryogenic treatment.

3.3 Wear Test

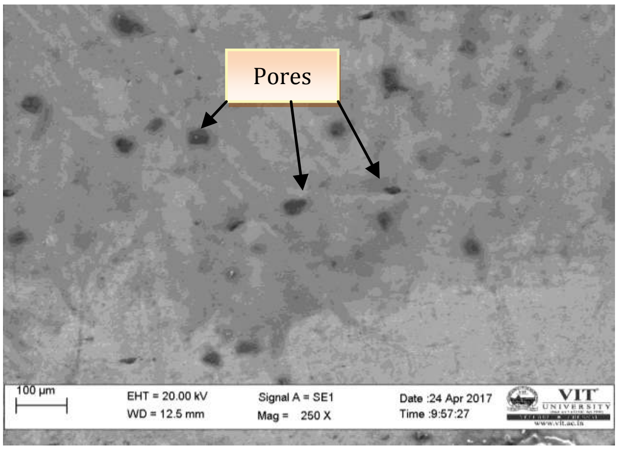
The effect of the cryogenic treatment on the AM 316L SS in terms of wear resistance was studied using a reciprocating wear test. The variation of the friction coefficient over the sliding time can be seen in Fig. 6.

Fig. 6. Coefficient of friction versus time plot for additive manufactured 316L SS before and after cryogenic treatment.

The Coefficient of Friction (COF) plot for both specimen displays three distinct phases, namely i) running-in period ii) transition period and iii) steady state period. COF vs Time plot displays similar behavior between specimen before and after cryogenic treatment during the initial running-in–period followed by a relatively linear friction transition phase. In a transition phase, variation in the magnitude of COF between cryogenic treated and untreated samples was observed. A steady state period was achieved after 1000 s, where the friction coefficient curves exhibit low and high friction plateaux. Moreover, the frequency of the transition from one regime to another over the sliding time was seen to be same for both untreated and cryogenic treated samples. Coefficient of friction for untreated sample was 0.41 and for cryogenic treated sample was 0.32. The observed friction coefficient evolution with time for cryogenic treated and untreated sample especially in the transition region represented changes in the wear mechanism. Some researchers have reported a change in wear mechanism in cryogenic treated conventional processed 316L SS as a result of the formation of the martensite state from austenite state without any change in chemical properties [35]. However, the mechanism behind the change in wear behavior for additive manufactured samples was investigated using surface roughness and wear track SEM analysis.

3.4 Wear Track and SEM analysis

The wear track of the AM 316L SS before and after cryogenic treatment was investigated using SEM analysis to understand the wear mechanism. Untreated 316L SS AM sample had a large number of micro-pores as shown in Fig. 7. a, whereas Micro pore presence was greatly reduced in the cryogenic treated sample (see Fig. 7b). Strain-induced severe plastic deformation of a specimen due to low temperature treatment could be phenomenon behind the sealing of pore in cryogenic treated AM sample.



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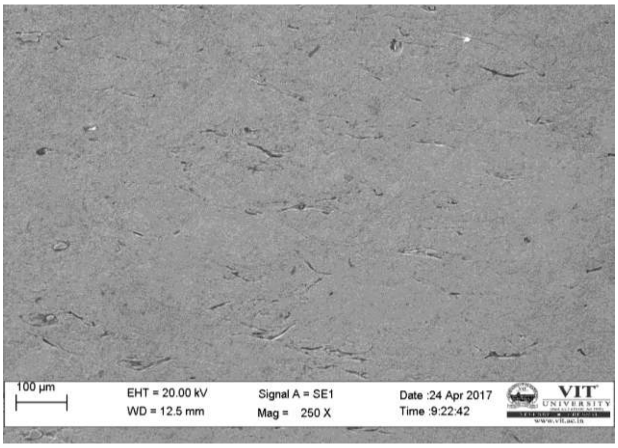
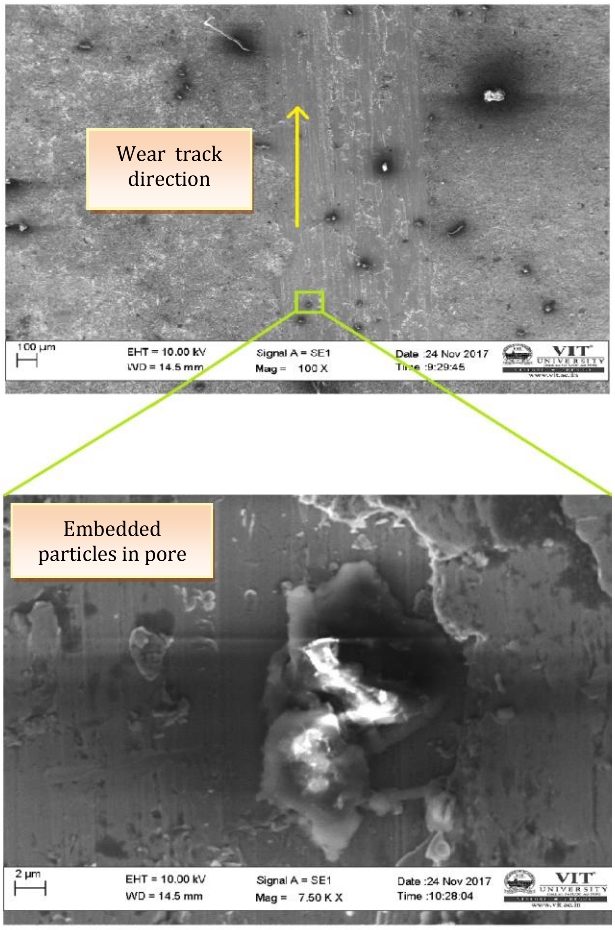


b)

Fig. 7. SEM image of additive manufactured 316L SS sample a) before and b) after cryogenic treatment.

SEM micrograph (Figs. 8a and 8b) highlighted the plastic deformation where the flow lines along sliding directions was shown in yellow arrow marks and some micro-cracks representing that the surface had undergone high contact stresses during sliding. From the SEM observation, it can be concluded that during the initial period of wear test, powder particles start to disintegrate from the sintered disc material, i.e. an additive manufactured 316L SS, during which there was a rapid increase in the friction coefficient, an accommodation was the surface of the first body that was more ductile. Since the particulate body and the specimen was of the same material and ductile in nature, there were severe deformation and plastic flow in wear track as observed, which are characteristics of adhesive wear [36]. Wear debris was seen embedded into the porosity present in the wear track of the untreated AM sample, whereas it was absent in cryogenic treated sample wear track due to absence of porosity in it. The wear track displayed different form of surface morphology between before and after cryogenic treated AM sample, due to this phenomenon.



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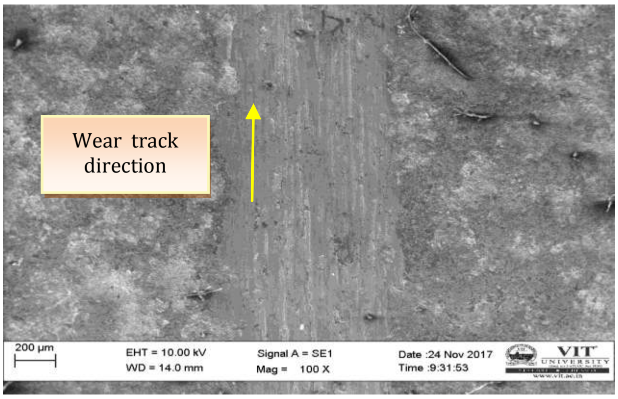
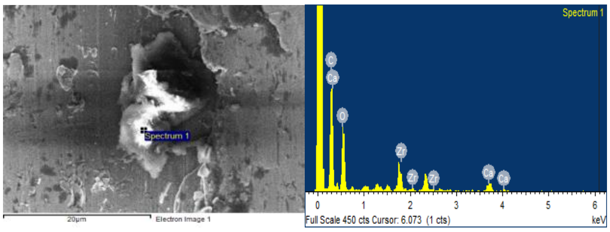


b)

Fig. 8. SEM image of additive manufactured 316L SS specimen wear track a) before and b) after cryogenic treatment.

The presence of loose wear debris increases the effective contact area between the ball and specimen which results in a more contact pressure and a more severe wear of the untreated compared with cryogenic treated sample [37]. This causes a significant increase in the magnitude of COF in the transition phase of untreated AM sample. The sliding of the ball against the disc promotes a strong temperature rise in the sliding surface, leading to the preferential oxidation of the surface.



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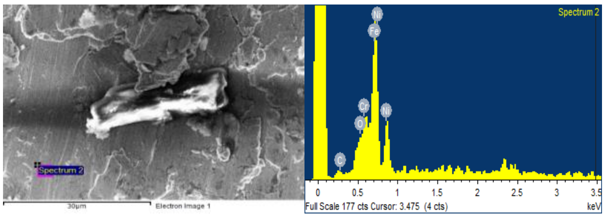


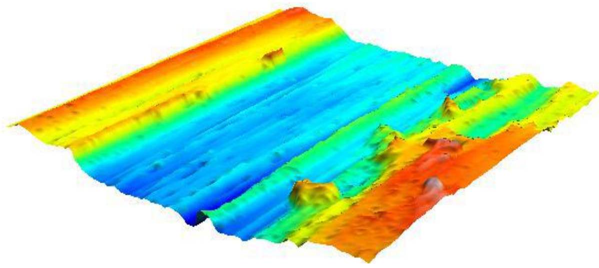
b)

Fig. 9. EDAX analysis of a) particle present in wear track and b) wear track of untreated additive manufactured 316L SS sample.

EDAX analyses of wear track and particle present in wear track are shown in Fig. 9 displays the presence of oxygen arising as a result of a rise in temperature between the sliding components. In addition, the presence of Zirconia in the debris of wear surface indicating the transfer of material from the ball to the 316L SS AM sample [38]. Wear track predominantly has ferrous, Chromium and Nickel. A layer of metal and oxidized wear debris was formed on the wear track, resulting in formation of fragmented oxide as a third body in wear track [39]. These abrasives stabilize the friction coefficient in the third phase of COF with respect to time (see Fig. 6).

3.5 Wear Track Surface Characteristics

The Surface morphology of the untreated and cryogenic treated AM 316L SS sample wear track is shown in Figs. 10a and 10b. The wear surface of the untreated AM samples shows the presence of irregular morphologies of the wear grooves along the track lines, with clear evidence for the indentation of the abrasives in pores.



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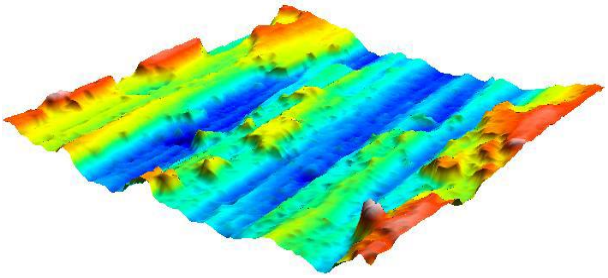
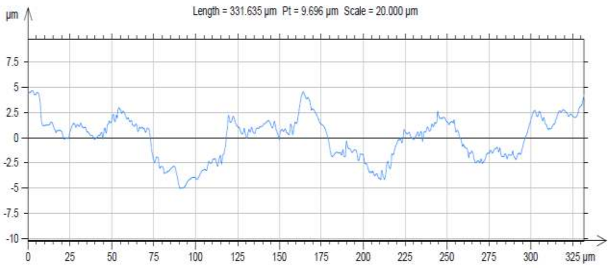


b)

Fig. 10. 3D Roughness profile of the wear track in a) untreated & b) cryogenic treated additive manufactured 316L SS sample.

The wear track of cryogenic treated AM sample shows comparatively smooth and shallow compared to the wear tracks of the untreated AM sample which displayed large peaks and valley (Fig. 10a), these observations indicating the occurrence of the reduced wear in cryogenic treated AM sample. The Average roughness profile of the wear track for before and after cryogenic treated AM sample is shown in Fig. 11. Roughness parameters of samples wear surface track are summarized in Table 3. Average Roughness (R_a) of the untreated AM sample was slightly higher than cryogenic treated AM sample.



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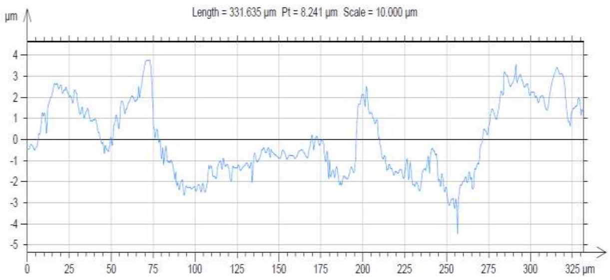

b)

Fig. 11. 2D Roughness profile of wear surface in a) untreated & b) cryogenic treated additive manufactured 316L SS sample.

Table 3. Roughness parameters of additive manufactured SS 316L before and after cryogenic treatment.

Parameter	Before Cryogenic Treatment	After Cryogenic treatment
Average Roughness (Ra) in μm	0.98	0.71
Kurtosis of the roughness profile (Rsku)	4.33	2.56
Material volume (μm3/μm2)	0.12	0.06
Void Volume (μm3/μm2)	5.09	4.71

Presence of high wear resistance strain induced martensite and the absence of pores resulted is a reduction of the peak width and peak height in cryogenic treated AM sample. Kurtosis of roughness profile was less than 3 for cryogenic treated sample, whereas, in the case of untreated AM sample it was greater than 3, this observation indicates the presence of sharp peaks and valleys in untreated AM sample. Further, roughness parameters in terms of material volume and void volume were higher in untreated AM 316L SS sample. From these observations it was concluded that the material removal is higher during wear test in the AM 316L SS sample before cryogenic treatment.

4. CONCLUSION

This study is an investigating the effect of cryogenic treatment on the wear behavior of additive manufactured 316L SS material. Based on the characterization of cryogenic treated samples, following conclusions have been drawn.

- Micro Vickers hardness was observed to experience a significant increase of 28.54 % post cryogenic treatment.
- Microstructure of Additive manufactured 316L SS is distinct from the conventional processed 316L SS.
- The microstructure study and SEM analysis show post cryogenic treatment resulting in the minimization of porosity due to overall shrinkage of specimen.
- Residual stress analysis using XRD pattern reveals the phase change in cryogenic treated specimen and there is a 34.54 % increase in compressive residual stress.
- There is a 17 % decrease in the coefficient of friction in cryogenic treated sample.
- Adhesive component, oxidative wear and abrasive wear mechanisms are observed in Tribology studies on AM sample.
- Insignificant change is seen in average surface roughness for post cryogenic treatment.
- Thus, the study conducted, shows improvement in hardness and decreases in wear for cryogenic treated AM samples, indicating the scope for cryogenic treatment as one of the potential post processing technique for additive manufacturing process.

Acknowledgements

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