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M. Hardt
RWTH Aachen University, Germany

F. Klocke
RWTH Aachen University, Germany

B. Döbbeler
RWTH Aachen University, Germany

M. Binder
RWTH Aachen University, Germany

Ibrahim S. Jawahir
University of Kentucky, is.jawahir@uky.edu

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Experimental study on surface integrity of cryogenically machined Ti-6Al-4V alloy for biomedical devices

M. Hardt[a,*], F. Klocke[a], B. Döbbeler[a], M. Binder[a], I.S. Jawahir[b]

[a]WZL of the RWTH Aachen University, Campus-Boulevard 30, 52074 Aachen, Germany
[b]ISM of the University of Kentucky, 143 Graham Ave, Lexington, KY 40506, USA

* Corresponding author. Tel.: +49 (241) 80-28174; fax: +49 (241) 80-22293. *E-mail address:* m.hardt@wzl.rwth-aachen.de

Abstract

Titanium and its alloys are widely used in the biomedical sector. In this field, titanium and its alloys are the material of choice for biomedical devices such as hip and knee replacements. Usually, a Total Hip Replacement (THR) is based on four components, made out of different materials due to the material properties associated with the functional performance. One approach to lower the overall manufacturing costs and enhance the reliability of THR's is to manufacture the prosthesis out of one material. The titanium alloy Ti-6Al-4V is, therefore, feasible as it exhibits better osseous integration compared to other metallic materials used as orthopedic devices. The sole use of Ti-6Al-4V alloy requires improvements of surface integrity (SI) and characteristics that are sensitive to SI. One possible way to improve the tribological properties of the THR and the biocompatibility of Ti-6Al-4V alloy is to deliberately decrease the material grain size in the surface layer from the micron scale (> 1 µm) to the region of nano-sized grains (< 100 nm).
The objective of this paper is to study and prove the formation of nano-sized grains within the surface as well as the characterization of surface integrity when machining Ti-6Al-4V alloy. Therefore, different cryogenic cooling strategies are used where liquid nitrogen (LN_2) is applied to the flank and rake face, and just to the flank face respectively. To compare the effect of cryogenic machining, conventional flood cooling was applied as third cooling strategy. As cutting tool, a roughing tool, having a large cutting edge radius, was used, since severe plastic deformation (SPD) has shown to be capable to produce nano-sized grains in the surface. The results showed, that cryogenic machining using a large cutting edge radius tool is able to decrease the materials grain size to the region of nano-sized grains.

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Keywords: Cryogenic machining; Ti-6Al-4V alloy; Grain refinement

1. Introduction

Titanium alloys are widely used in the medical sector, where they have been used as replacement for teeth (dental implants) and bone (maxillofacial devices, components of hip and knee replacements) as well as for stents [1,2]. The wide acceptance and application of titanium alloys is due to their unique material properties. The main advantage of this class of material is the high strength to density ratio [3]. For the usage as biomaterial, properties such as the excellent corrosion resistance as well as the good biocompatibility make this group of material the material of choice when replacing hard tissue [4,5]. Compared to other biomaterials such as Al_2O_3 or CrCo-alloys, the modulus of elasticity of titanium alloys is low. A low modulus of elasticity is favorable, since it is desired to be similar to that of the human hard tissue (bone) [6].

Among medical implants to replace impaired hard tissue, the hip replacement is one of the most challenging devices in terms of manufacturing [2]. Usually, a Total Hip Replacement (THR) is based on four different components: the femoral stem, femoral head, liner, and acetabular cup [7]. Due to different challenges concerning the material properties associated with functional and operational requirements where the basic components of a THR are made of different materials. The femoral stem is made out of titanium alloys

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such as Ti-6Al-4V due to the excellent in vivo corrosion resistance and strong osseous integration [4]. The femoral head on the other side is made out of ceramics such as Al_2O_3 or ZrO_2 due to favorable wear and tribological properties. The femoral head rotates in a mostly metallic acetabular cup, separated by a liner made out of ultrahigh molecular weight polyethylene (UHMWPE) [2].

One approach to lower the overall manufacturing costs and enhance the reliability of hip replacements is to manufacture the prosthesis out of one material, providing the opportunity to reduce the number of parts. Titanium alloys, such as Ti-6Al-4V, are therefore favorable as they exhibit a better osseous integration compared to other metallic materials used as orthopedic devices. The overall usage of the titanium alloy requires an improvement in material properties, especially concerning the wear and tribological behavior.

A feasible way to improve these properties is to decrease the materials grain size in the surface layer from the micron scale (> 1 µm) to the region of nano-sized grains (< 100 nm) [8,9,10]. For other metallic materials, such as the magnesium alloy AZ31B, cryogenic machining with a large cutting edge radius has shown to be a suitable manufacturing process for grain refinement [11,12].

The objective of this paper is to study and prove the formation of nano-sized grains within the surface layer as well as the characterization of surface integrity (SI) when machining Ti-6Al-4V alloy using a roughing tool, finishing machining parameters, and cryogenic cooling conditions.

2. Experimental set-up and procedure

The studied work material was the commercial titanium alloy Ti-6Al-4V. The material has been received from VSMPO-AVISMA in the form of a 3.3 mm thick sheet. The test specimens, having a diameter of 114.4 mm were cut from the sheet. The materials nominal chemical composition is shown in Table 1. The materials bulk hardness was measured to be 350 HV0.1.

Table 1. Nominal chemical composition of Ti-6Al-4V alloy in mass-%.

Element	Al	Fe	O	Ti	V
Mass percentage	6	< 0.25	< 0.2	Rest	4

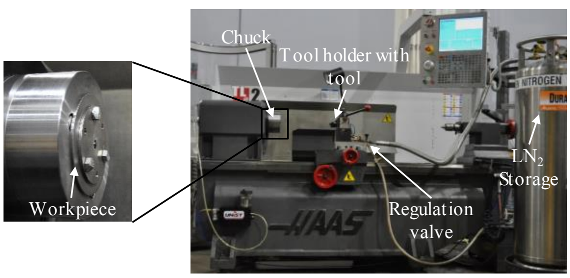

Fig. 1. Experimental set-up of the conducted face turning trials.

Face turning trials were conducted on a computer numerical controlled Haas TL-2 horizontal bed lathe at the Institute of Sustainable Manufacturing (ISM) of the University of Kentucky. The experimental set-up is shown in figure 1.

Since high strains and strain rates have shown to be an actuator for dynamic recrystallization (DRX) [13] which can lead to the process of grain refinement [14], a cutting tool with a large cutting edge radius was used. Thus, ploughing was benefited causing higher strains and strain rates within the surface of the samples. As tool, indexable inserts made out of cemented carbide were used. To avoid the influence of tool wear, the cutting edge was changed after every cut with a feed travel of $l_f = 20$ mm. The rounded cutting edge radius was measured for each indexable insert at three different spots using a Zygo NewView 7300 white light interferometer to $r_\beta = 59$ µm with a standard deviation of 6 µm. The working clearance angle of the cutting tool was according to the tool holder $\alpha_{0e} = 7°$.

As cooling conditions, cryogenic coolant using liquid nitrogen (LN_2) was applied to either just the flank face or to both, flank and rake face. The LN_2 was stored in a Dura-Cyl Liquid Cylinder dewar. The LN_2-jet was pumped from a 1.03 MPa (150 psi) pressurized tank. Cryogenic coolant was used since it is beneficial for grain refinement due to an increase of the materials hardness and therefore leading to higher plastic deformation [15] as well as due to the suppressing of grain growth occurring during high temperatures (annealing) [11].

To set a base-line, the two cryogenic cooling conditions were compared to conventional flood cooling since flood cooling represents the state of the art when machining Ti-6Al-4V alloy. The set-up of all three cooling conditions is shown in figure 2.

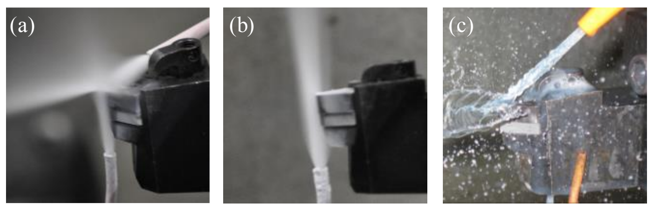

Fig. 2. Set-up of the applied cooling strategies: (a) cryogenic flank and rake face cooling, (b) cryogenic flank face cooling; (c) conventional flood cooling.

The cutting parameters were set according to the state of the art when machining Ti-6Al-4V alloy and are summarized in table 2 [16]. To evaluate different process parameters and their influence on SI, a full factorial test matrix was chosen. In total 24 machining trials were conducted.

Table 2. Parameters of the conducted machining trials.

Process parameter	Value of the process parameter			
Corner radius r_ε [mm]	1.6			
Depth of cut a_p [mm]	0.2			
Cutting speed v_c [m/min]	75		150	
Feed rate f [mm]	0.050	0.075	0.100	0.125

3. Experimental results

3.1. Roughness

Among the parameters of SI the surface roughness is considered to be the primary indicator of the quality of the machining process since it influences the functional performance of the machined workpiece [17]. In terms of the osseous integration, the surface roughness has shown a strong influence on the ingrowths of biomedical devices, due to an improved bioadhesion and an increased cellular attachment [2, 18].

The surface roughness of the machined samples was measured using a Zygo NewView 7300 white light interferometer. For each sample the roughness Ra was measured on three different spots, whereby roughness measurements were performed on each spot 10 times in the direction of feed on a length of 0.435 mm. The results of the surface roughness measurements when machining with a cutting speed of v_c = 75 m/min are shown in figure 3.

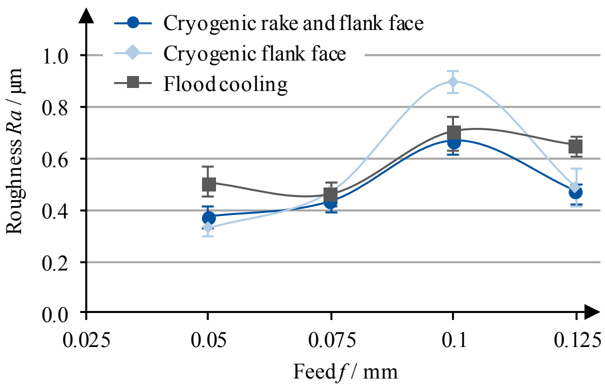

Fig. 3. Surface roughness *Ra* for the three cooling strategies when turning with a cutting speed of v_c = 75 m/min.

When turning with a cutting speed of v_c = 75 m/min the smallest surface roughness can be found for the cryogenic cooling conditions and the smallest feed rate. The surface roughness measurements for all three cooling conditions show a comparable development when increasing the feed rate and are all within the same magnitude smaller than Ra = 1.0 µm.

An increase of the feed rate from f = 0.05 mm to f = 0.1 mm leads to an increase of the surface roughness, whereas a further increase of the feed rate to f = 0.125 mm leads to a decrease. The decrease of the surface roughness can be explained by thermal softening, caused by the higher feed rates and leading to a smoother surface.

In comparison figure 4 shows the surface roughness for all three cooling conditions for a cutting speed of v_c = 150 m/min. It shows comparable results for all three cooling strategies. An increase of the feed rate up to f = 0.1 mm leads to an increase of the surface roughness, whereas a further increase up to f = 0.125 mm leads to a stagnation or slightly decrease of the surface roughness.

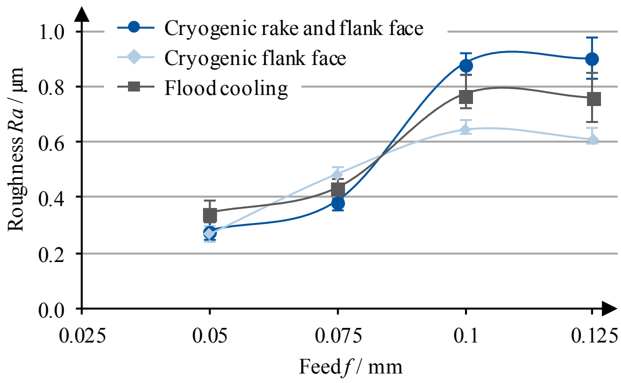

Fig. 4. Surface roughness *Ra* for the three cooling strategies when turning with a cutting speed of v_c = 150 m/min.

When comparing the topographical images of the machined samples for the three different cooling strategies it can be concludes that different types of material damages caused by the machining process occur [8], figure 5. The topographies of the cryogenic machined samples show a surface that was partly ploughed (a, b), whereas the flood-cooled counterpart (c) shows a topography with periodic peeks and valleys, indicating cutting instead of partly ploughing.

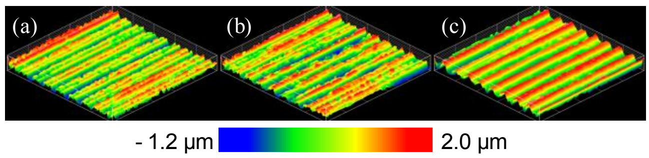

Fig. 5. Surface plot (0.435 mm x 0.435 mm) of the samples, v_c = 75 m/min, f = 0.05; (a) cryogenic flank and rake face cooling; (b) cryogenic flank cooling; (c) flood cooling.

These observations support the hypotheses that significant lower temperatures occur during the cryogenic cooling compared to the conventional flood cooling. The lower temperatures lead to an increase of the materials strength, impairing the machinability and promoting ploughing.

3.2. Hardness

To measure the effect of the machining parameters and the applied cooling conditions on the materials hardness, two kinds of hardness measurements were performed on a Sun-Tec CM-800 Vickers tester according to DIN EN ISO 6507-1. An indention force of F = 0.1 kp (= 0.98 N) and an indention time of t = 15 s were used. For one thing, hardness measurements were done on the surface of the machined samples, for another thing hardness measurements were done along the direction of depth of the machined samples. For both types of hardness measurement, five indents were performed in order to receive a surface hardness value or a hardness value in a certain distance to the machined surface.

The results of the surface hardness measurements are summarized in figure 6 (a) for a cutting speed of v_c = 75 m/min and in (b) for a cutting speed of v_c = 150 m/min. As it can be seen in figure 6 (a) all three cooling conditions are capable to increase the surface hardness for a cutting speed of $v_c = 75$ m/min. The largest increase of hardness can be observed for the cooling condition, where LN_2 was applied just to the flank face. Therefore, the increase of the surface hardness is 7.0 % for a feed rate of $f = 0.05$ mm and 12.5 % for a feed rate of $f = 0.125$ mm, respectively.

On the other side, figure 6 (b) shows that for a cutting speed of $v_c = 150$ m/min only cryogenic cooling conditions are capable to increase the surface hardness, whereas flood cooling leads to a decrease. For the higher cutting speed the hardness of the cryogenic machined samples increased even more than it did for the lower cutting speed. The highest increase of the hardness was achieved, when the cryogenic coolant was applied only to the flank face. In this case the surface hardness increased by 16.5 %.

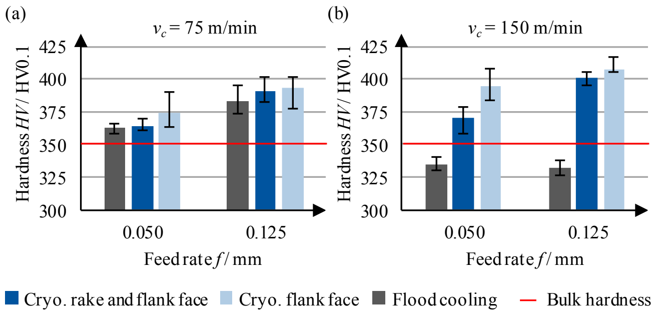

Fig. 6. Influence of the cooling conditions on the surface hardness: (a) $v_c = 75$ m/min, (b) $v_c = 150$ m/min.

The larger increase of the surface hardness for the cooling condition where LN_2 was just applied to the flank face can be explained by the flow rate of the coolant. Since the same tank pressure was used for both cryogenic cooling conditions, the double amount of LN_2 was applied to the materials surface when cooling just the flank face compared to cooling both, flank and rake face.

Besides an evaluation of the influence of the cooling conditions on the surface hardness, the influence of the cutting parameters feed rate and cutting speed was evaluated. Therefore, surface hardness measurements of all samples where LN_2 was applied to the flank and to the rake face were conducted, figure 7. For both, increasing the cutting speed and increasing the feed rate, an increase of the surface hardness was observed. Doubling the feed rate from $f = 0.05$ mm to $f = 0.1$ mm leads to an increase of the surface hardness of 4.8 % for $v_c = 75$ m/min and of 3.9 % for $v_c = 150$ m/min, respectively. On the other side, doubling the cutting speed from $v_c = 75$ m/min to $v_c = 150$ m/min leads to a maximal increase of 2.7 % of the surface hardness.

To analyze the development of the hardness in the direction of depth of the machined surface, hardness measurements were done within a distance of 25 µm within the first 100 µm, and of 50 µm within the following 300 µm, figure 8. As shown, the materials bulk hardness is reached within 100 µm distance to the machined surface. Comparable results were observed for the other machining parameters being analyzed.

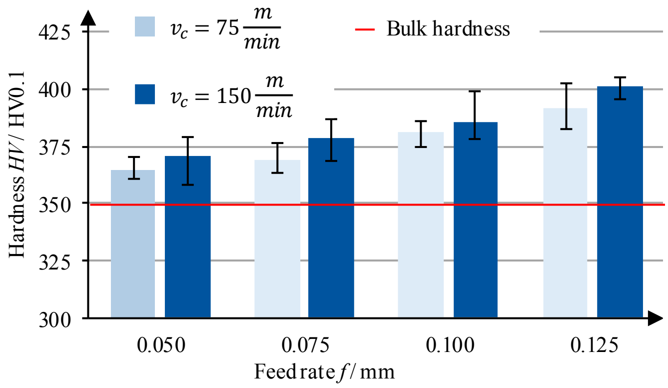

Fig. 7. Influence of the cutting parameters on the surface hardness for cryogenic flank and rake face cooling.

As shown in the literature [15], it can be concluded, that cryogenic machining of Ti-6Al-4V alloy is a capable way to increase the hardness of the surface layer. The materials temperature behind the cutting zone, which is related to the amount of coolant, showed to influence the materials hardness. This is due to the increased materials' strength, causing more severe plastic deformation and work hardening from the machining process. On the other side, the cooling effect of conventional flood cooling is not high enough to suppress thermal softening due to annealing for the higher cutting speed of $v_c = 150$ m/min.

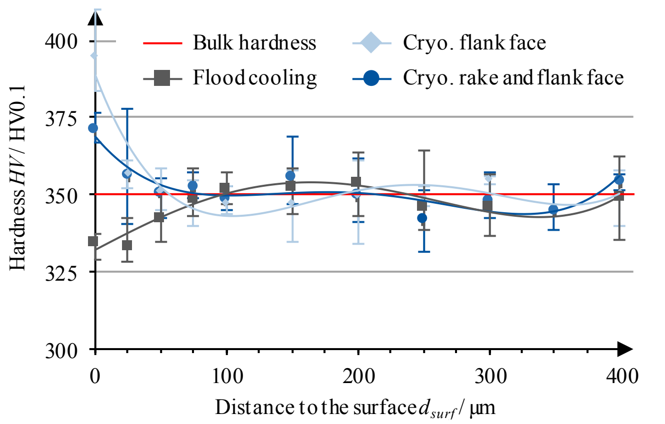

Fig. 8. Micro hardness measurements for three different cooling strategies when turning with $v_c = 150$ m/min, $f = 0.05$ mm.

3.3. Microstructure

The influence of the machining parameters on the microstructure of the machined surface was evaluated by measuring the thickness of the effected layer. Therefore, the thickness of the effected layer of polished and etched samples was measured using an optical microscope. The results are shown in figure 9.

The measurements show that the thickness of the effected layer is the largest when cooling with LN_2 just on the flank face. The cryogenic counterpart where the LN_2 is applied to flank and rake face causes just slightly thinner layers. In case of conventional flood cooling, the thickness of the effected layer is within the same magnitude compared to the cryogenic cooling condition when turning with a cutting speed of v_c = 75 m/min. Increasing the cutting speed to v_c = 150 m/min leads to slightly thinner layers than it does for the cryogenic counterparts.

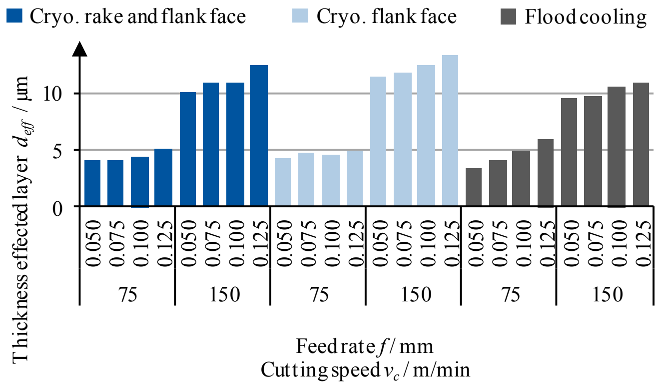

Fig. 9. Thickness of the effected layer dependent on the machining parameters and the cooling conditions.

The reason for the thicker effected layer of the cryogenic machnined samples is expected to be due to the significant lower temperatures occuring during the cutting proecess. The lower temperatures cause the materials strength to increase and therefore, causing more SPD. Among the analyzed cutting parameters, the cutting speed has the largest influence on the thickness of the effected layer, whereas the feed rate has a minor influence.

3.4. Nanostructure

Nanostructure measurements were done in order to verify or falsify the hypotheses that cryogenic machining of Ti-6Al-4V using a roughing tool with a large cutting edge radius and finishing machining conditions is capable to decrease the materials grain size in the surface layer to the region of nano-sized grains. To reveal the grain structure within the surface layer scanning electron microscopy (SEM) and focused ion beam (FIB) images using the field emission gun (FEG) Helios Nanolab 660 were taken. Due to high costs, just one sample was analyzed. The sample that was analyzed was machined under cryogenic conditions (flank and rake face) with a cutting speed of v_c = 75 m/min and a feed rate of f = 0.05 mm.

The grain size was measured using the public domain software ImageJ. The diameter of a single grain was measured in four different directions, having an angle of 45° to each other. The grains were measured along five parallel lines, having a distance of 5 nm, 250 nm, 1 µm, 3 µm, and 5 µm to the surface. For each of the distances 10 grains were measured.

Within the surface layer of the machined sample, three different regions can be identified, figure 10. At the top most, grains having an average grain size of 28 nm can be found. The average grain size increases up to 67 nm within a distance of 250 nm, and to 103 nm within a distance of 1 µm. Therefore, the grains of this refined layer, that has a total thickness of 1 µm, can be classified as nano-sized grains, since their average grain size is smaller than 100 nm.

The transition zone can be found between the refined layer and the bulk material. Within this zone, grains having a distance of 3 µm to the machined surface were measured to have an average grain size of 900 nm and can therefore be classified as ultrafine grains (UFG) [20]. In a further distance of 5 µm to the surface the materials bulk grain size is reached. The bulk grain size was measured to be 2.5 µm. Thus, the transition zone has a total thickness of 4 µm and the total thickness of the effected layer is about 5 µm. Hence, the measurement of the effected layer on the nano scale meets the measurement that was done on the micron scale.

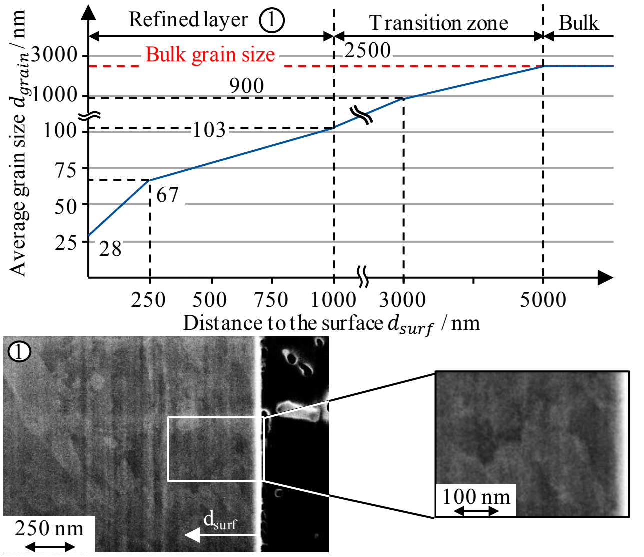

Fig. 10. Average grain size over the distance to the machined surface (cryogenic flank and rake face cooling, f = 0.125 mm, v_c = 75 m/min).

4. Conclusion

The presented paper focuses on the effect of machining Ti-6Al-4V alloy under cryogenic conditions using a cutting tool with a large cutting edge radius and its influence on the surface integrity. Therefore, the surface roughness as well as the materials hardness, micro-, and nanostructure were analyzed. The key findings can be summarized as follows:

- The surface roughness of all machined samples was found to be smaller than Ra = 1 µm. The smallest surface roughness was found to be Ra = 0.27 µm. Hence, machining with a roughing cutting tool and finishing machining conditions is capable to produce surfaces which are comparable to grinded surfaces, based on the surface roughness.
- The surface hardness of the cryogenic machined samples was found to be higher than the materials bulk hardness. An increase up to 16.5 % was measured. Hence, this process variant may be capable to obviate potential post processes to increase the materials hardness. It has been shown that high severe plastic deformation, caused by higher feed rates and higher cutting speeds, as well as cryogenic cooling leads to the highest increase of the materials hardness.

- The measurement of the thickness of the effected layer on the nano scale using a FIB-analysis meets the results of the measurement on the micron scale. Therefore, a good agreement for the samples that were not analyzed on the nano scale can be expected.
- Cryogenic machining of Ti-6Al-4V alloy using a large cutting edge radius tool and finishing machining conditions is capable to reduce the materials grain size within the surface layer down to the region of nano-sized grains (< 100 nm).
- Among the two studied cryogenic cooling conditions, cryogenic cooling of just the flank face has shown to lead to the largest increase of the hardness and of the thickness of the effected layer. This observation can be explained by the mass flow rate of LN_2. Compared to cooling flank and rake face with LN_2 the mass flow rate was about the double when just cooling the flank face. This difference of the mass flow rate causes lower material temperatures when just cooling the flank face. Consequently, the lower temperatures lead to an increase of the materials strength and hence, to more severe plastic deformation during the machining process.

In summary, cryogenic machining of Ti-6Al-4V alloy under cryogenic cooling conditions using a large cutting edge radius tool has shown to be capable to decrease the materials grain size within the surface layer to the region of nano-sized grains. Since nano-sized grains have shown to decrease the run-in time [21] as well as to enhance the cell compatibility with (pre-) osteoblasts [22,23,24], this process variant may be an innovative way to enhance the reliability of THR due to an improvement of the surface integrity.

Acknowledgements

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