Investigation on the Effect of Low Temperature on Impact Properties of Ti-6Al-4V Titanium Alloy

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Abstract
Cryogenic cutting is becoming an attractive machining method for difficult-to-cut materials. However, it’s very difficult to analyze directly their cutting mechanism at low temperature. In order to better understand the various physical phenomena in the cryogenic cutting of titanium alloy, the Charpy impact test of Ti-6Al-4V titanium alloy at low temperatures (as low as -196 °C) was undertaken in this work. The Charpy absorbed energy of Ti-6Al-4V titanium alloy at low temperatures was investigated firstly. Then, by observing the microscopic and macroscopic morphology of the fracture, the impact properties and fracture modes of Ti-6Al-4V titanium alloy at low temperatures were analyzed. It was found that the impact toughness of Ti-6Al-4V titanium alloy reduces when the temperature decreases from 20 °C to -196 °C, and the fracture appears a tendency to become brittle. Meanwhile, three kinds of areas, i.e. shear lip area, fiber area, and radiation area, were found on the fracture morphology at each temperature. Those areas correspond to the shear fracture zone, crack initiation zone, and crack extension zone, respectively. With the decrease in temperature, the proportion of fiber area decreases, and the radiation area appears and increases gradually. However, fiber areas were still observed on the macroscopic morphology of the fracture under – 196 °C, which suggests that Ti-6Al-4V titanium alloy still has the ability to deform plastically at such low temperatures. The research result in this work provide a fundamental support for analyzing the cutting mechanism of Ti-6Al-4V titanium alloy at low temperatures.

1. Introduction
Titanium alloys have excellent comprehensive mechanical properties: high specific strength, corrosion resistance, high strength at high temperatures and so on. Therefore, titanium alloys have been widely used in industries such as aerospace, shipbuilding, and motor racing [1] [2]. However, titanium alloys are typical difficult-to-cut materials, which have high requirements for the processing environment. Conventional processing methods are inefficient and the surface quality is uncontrollable. It is often necessary to use cutting fluids [3]. Whereas, cutting fluids is generally not an environment-friendly coolant and lubricant, as is known to all.

At present, cryogenic manufacturing process has been a hot research topic in the field of mechanical
manufacturing, and it has always been regarded as a kind of clean manufacturing technology. Researchers have been investigating impact of cryogenic machining with liquid nitrogen (LN$_2$) on machining performance in both academia and industry. Ramli et al. [4] and Sartori et al. [5] used nitrogen gas and LN$_2$ as coolants to machine titanium and its alloys, then they compared it with dry machining. The authors reported that machining with LN$_2$ had excellent advantages for tool wear and machined surfaces quality. Shokrani et al. [6] performed milling tests on the Ti-6Al-4V titanium alloy. From comparison with dry and wet conditions, it was found that cryogenic machining not only reduced the surface roughness of the machined parts but also prolonged the tool life. Similarly, Jawahir et al. [7] also conducted experiments on cryogenic machining. It was found that cryogenic machining can obtain better surface quality while reducing or eliminating damage caused by heat generated by the process. Biermann et al. [8] conducted the experiments of two kinds of titanium alloys. In their study, when machining Ti-6Al-4V titanium alloy with low temperature CO$_2$ as coolant, the tool life could be extended compared with emulsion. When machining high strength titanium alloy Ti-6Al-2Sn-4Zr-6Mo, an additional lubrication mechanism was required to get better processing quality. Additionally, Trabelsi et al. [9] focused on the effect of cryogenic assistance on tool life when machining Ti17 titanium alloy, and found tool life was prolonged but temperature had little effect on cutting force. A recent study done by Zhao et al. [10] [11] also reported that the cryogenic cooling (LN$_2$ cooling) has a significant influence on the chip formation, cutting force, and surface integrity of Ti-6Al-4V titanium alloy. However, it was found from the above literatures that the analysis on the cryogenic cutting mechanism seems still insufficient enough due to the less of measurement data on the material properties at low temperature. Analyzing the cryogenic properties of materials is very important to the study of cryogenic machining. Many researchers have studied the mechanical properties of titanium alloy materials at low temperature. As early as 1974, Campbell [12] found that the effect of temperature on the toughness of alloy materials depended on the alloy matrix. As the temperature decreases, titanium alloy generally had lower toughness, but it was affected by alloy content and the way of heat treatment.
Some titanium alloys still maintained good toughness at low temperature. Subsequently, Moskalenko et al. [13] systematically studied the deformation mechanism of pure titanium and its alloys at low temperature, and found that the plasticity variation of pure titanium and its alloys at 40 ~ 120 K was due to combined action of two deformation mechanisms, i.e. slip and lower temperature activated twinning. Hong et al. [14] studied the low-temperature mechanical properties of Ti-6Al-4V titanium alloy and found that its strength and hardness increased with the decreases of temperature, but the plastic toughness did not change significantly with the decrease of temperature. Sun et al. [15] conducted tensile tests and low-cycle fatigue tests at 293K and 77K for Ti-2.5Cu titanium alloy, and found that titanium alloy had higher ductility and longer low- cycle fatigue life at 77K than that at 293K. Ono et al. [16] studied the performance of Ti-5%Al-2.5%Sn at low temperature and found that the ultimate tensile strength decreased with decreasing temperature, and the temperature had little effect on the crack initiation of the specimen. Bertolini et al. [17] compared the changes in material properties of titanium alloy under conventional parameter processing and low-temperature processing conditions. Furthermore, the morphology of fractures at low temperature was also analyzed. The results suggested that temperature and strain had a significant influence on material properties. In addition, Semenova et al. [18] conducted tensile tests and Charpy tests to study the influence of temperature on ultrafine-grained Ti-6Al-4V titanium alloy, and investigated that the ultimate tensile strength and yield stress enhanced in a wide range of temperature, and the absorbed energy decreased when the temperature reduced.

As evident from the above literatures review, the impact resistance is an important performance index of the material. In consideration of the high-frequency impact characteristics in the process of cutting, the impact test does help to investigate the cutting mechanism, especially to analyze the influence of cryogenic cooling on cutting process. At present, there are already studies on the impact properties of titanium alloys under low temperatures. However, the analysis of impact properties for Ti-6Al-4V titanium alloy under a relatively dense distribution of low temperatures (20 °C-196 °C) is rarely reported in the available literature. Therefore, the present work aims to analyze the impact properties of Ti-6Al-4V titanium alloy at low temperatures by investigating Charpy absorbed energy.
and fracture morphology at different temperatures. The investigations are focused on the effect of low temperature on the impact properties of Ti-6Al-4V titanium alloy, and expected to provide an positive guidance for investigation on the cryogenic cutting of titanium alloy and its cutting mechanism.

2. Experimental Setup
2.1 Material and equipment

The test material used in this investigation is a kind of annealed Ti-6Al-4V titanium alloy, which is a typical two-phase $\alpha + \beta$ titanium alloy, with aluminum as the alpha stabilizer and vanadium as the beta stabilizer. The chemical composition of this material is given in Table 1, and its metallographic structure is as shown in Fig. 1.

| Element | Ti   | Al   | V    | Fe   | C    | N    | H    | O    |
|---------|------|------|------|------|------|------|------|------|
| Content | The rest | 5.50 ~ 6.75 | 3.50 ~ 4.50 | $\leq$ 0.30 | $\leq$ 0.10 | $\leq$ 0.05 | $\leq$ 0.015 | $\leq$ 0.20 |

The dimension of standard specimen for Charpy impact test is 55mm × 10mm × 10 mm, and a 45° V-notch having a 0.25 mm tip radius was pre-processed at the center of each specimen. All the specimens were polished before testing, and their surface roughness (Ra) were less than 0.10 µm.

The principle of impact test is shown in Fig. 2. All the impact tests were performed on an MTS-SANS ZBC-300A pendulum impact tester, as shown in Fig. 3.

2.2 Experimental design

According to the previous research work, i.e. cryogenic cutting Ti-6Al-4V titanium alloy[11], seven temperature points were selected from the range of 20 °C ~-196 °C for the impact test, as shown in Table 2. In addition, three specimens were tested at each temperature point. The low-temperature environment was obtained by disposing an alcohol-liquid nitrogen mixture, i.e. mixing different volumes of liquid nitrogen and alcohol in a cryogenic Dewar bottle to prepare the mixture with an expected low temperature, where the liquid nitrogen was used directly for the − 196 °C cooling. After immersing in the mixture for 15 minutes, the specimen was taken out and fixed on the impact tester by quick-clamping, then the impact test was carried out. In order to minimize the influence of temperature rising on the impact energy, each round of the test was finished within 10 s ~ 15 s.
2.3 Characterization of fracture zone

After the impact test, the fracture zones of specimens were analyzed with a 3-dimensional surface micro-analyzing system LEICA DVM6 and a Scanning Electron Microscope S-3400. Generally, the typical ductile fracture of metals has three regions: fiber area, radiation area, shear lip area, as shown in Fig. 4. The relative size of the three regions generally reflects the toughness of the material. A scale coefficient is usually used to indicate the toughness of the material. For instance, the ratio of the radiation zone to the area of the other two zones is defined as $R$, and the temperature at $R = 1$ is the ductile-brittle transition temperature [19]. Under the same conditions with the exception of temperature, the smaller the value of $R$, the higher the toughness of the material. As to the brittle fracture of metals, the percentage of brittle fracture has the same physical meaning as $R$, which is the ratio of the radiation area to the fracture area. Ti-6Al-4V titanium alloy is a typical thermo-viscoplastic material. Therefore, the same analyzing method was used to characterize the fracture zone in this work.

3. Results And Discussion
3.1 Effect of low temperature on impact energy

Generally, the material changes from plastic to brittle with the temperature dropping, and the impact absorbing energy will decrease significantly. The fracture will change from micro-void coalescence fracture to trans-granular fracture, which is called cryogenic brittleness [20] [21].

After the impact test, the impact energy, i.e. impact absorbing energy and impact toughness, was measured and analyzed. Then an average value was taken from three specimens under the same low-temperature condition. A graph was then drawn to show the relationship between the impact energy and specimen temperature, as shown in Fig. 5.

It can be seen from Fig. 5 that with the decrease in temperature, the impact absorbing energy and impact toughness of Ti-6Al-4V titanium alloy show a downward trend. At the range of 20 °C ~ -160 °C, there is no obvious sudden drop zone on the impact absorbing energy - temperature curve, and the impact absorbing energy dropped by 29.3%, which indicates that under this temperature range, the

| Group | 1 | 2  | 3  | 4  | 5  | 6  | 7  |
|-------|---|----|----|----|----|----|----|
| Temperature/°C | 20 | 0 | -40 | -80 | -120 | -160 | -196 |

Table 2
Setup of temperature points
impact absorbing energy value of Ti-6Al-4V titanium alloy has relatively less obvious ductile-brittle transition characteristics, but the decline of impact absorbing energy also indicates the tendency of ductile-brittle transition. However, when the temperature drops to -196 °C, the impact absorbing energy has a significant drop zone, and rate of decline is 40.7%, which indicates that the material plasticity at this temperature point has decreased and the material tends to become more brittle. As to this phenomenon, Runchen et al. [22] investigated the fracture toughness of Ti60 alloy, and found the toughness of samples decreased when temperature reduced. Semenova et al. [18] also found the same phenomenon when they studied the Charpy absorbed energy of UFG Ti-6Al-4V alloy at different temperatures. Therefore, the ductile-brittle transition of Ti-6Al-4V titanium alloy under low temperature, as shown in Fig. 5, verifies that the low temperature leads to increasing of the mechanical properties (e.g. yield strength) of Ti-6Al-4V titanium alloy, thereby increasing the cutting force when milling it under low temperatures, as presented in Ref. [11].

3.2 Effect of low temperature on fracture morphology
3.2.1 Macroscopic morphology of the fracture
The macroscopic morphology of the fracture zone was observed on a 3-dimensional surface micro-analyzing system LEICA DVM6, as shown in Fig. 6, which illustrates the fracture morphology at different temperatures.

It can be seen from Fig. 6(a) to Fig. 6(g) that the macroscopic surface of the first few temperature points is undulating, the fracture morphology at -196 °C is relatively flat and smooth, and the color of the fracture zone is gray with shear lip area, fiber area and radiation area. As the temperature decreases, the shear lip area of the fracture becomes smaller significantly, and the proportion of the radiation area gradually becomes larger, which leads to a decrease in the plastic deformation zone. In addition, the energy required for cracks propagation decreases, i.e. the propagation of cracks is easier. The macroscopic features of the fracture zone are the same as the results reported by Semenova et al. [18]. When the temperature is higher (20 °C), it can be clearly seen that the proportion of the fiber area of the fracture is large. There are many transverse lines perpendicular to the direction of crack propagation, which are corrugated or concentric. The fiber area is surround by
the shear lip area, but the range of the radiation area is not obvious, and even the secondary fiber area appears at the bottom of the fracture zone, which is a plastic fracture zone formed by the unstable propagation of cracks during the ductile fracture. When the temperature decreases, the proportion of the fiber area reduces significantly, and the area of the radiation area increases. The fracture characteristics corresponds to the trend of impact energy changing with temperature (Fig. 5). It indicates that the decrease of temperature results in a reduction of the material’s toughness and an increase of its brittleness, thereby a drop in the impact energy.

The propagation of cracks is shown in Fig. 8. The test specimens at 20 °C, -40 °C, -120 °C and −196 °C were chosen for the analysis. The fracture shape at 20 °C and −40 °C is significantly more tortuous than that at the lower temperature of -120 °C and −196 °C, indicating that the cracks propagation is subject to large resistance and plastic deformation. Furthermore, the cracks propagation path at -196 °C is relatively flat, which indicates that the propagation of cracks is easy at lower temperature, and its impact toughness value is correspondingly low. The variation of crack directions from 20 °C to -196 °C, as shown in Fig. 8, indicates that the lower temperature leads to the Ti-6Al-4V titanium alloy separates in a more straight direction (load direction). It strongly supports from the mechanism that why the surface finish can be improved when milling this alloy under LN₂ cooling condition, as presented in Ref. [11].

3.2.2 Microscopic morphology of the fracture
Observation of the fracture morphology generally requires the microscope to have a maximum depth of focus and the widest possible magnification range as well as high resolution. Thus an S-3400N scanning electron microscope was used to observe the fractures’ microscopic morphologies of Ti-6Al-4V titanium alloy at different temperatures, as shown in Fig. 9.

The microscopic morphology of the fracture zone also confirmed the plasticity characteristics of Ti-6Al-4V titanium alloy. At room temperature of 20 °C (Fig. 9(a)), large and deep dimples can be seen in the microscopic morphology of the fracture, indicating that the resistance to crack propagation is large, and the material undergoes large plastic deformation before the fracture of the specimen, which corresponds to the maximum impact energy too. With the decrease in temperature, as shown
from Fig. 9(a) to Fig. 9(g), the dimples seem to become smaller, but the size of them is uniform. It indicates that the number of crack initiation is even more at low temperature. Similarly, the number of dimples decreases as the temperature drops, which also indicates a reduction on the plasticity of the material.

The micro-morphology of the fracture zone at -196 °C (Fig. 9(g)), as well as the occurrence of secondary cracks, presents a quasi-cleavage fracture with plastic and brittle mixing. It indicates that the characteristics of brittle fracture increases at this temperature. The cleavage plane is a microscopic platform formed by crack trans-granular fracture along a certain orientation plane, which is a typical brittle-dominantly fracture. With the decrease of temperature, the cleavage plane of the fracture gradually increases, some secondary cracks appear at the same time, and the fracture has been partially characterized by brittle fracture. What’s more, it can be found that the number of dimples in the fracture at -196 °C is higher than that at 20 °C, by comparing Fig. 9(a) with Fig. 9(g). However, the enhancement of absorbed energy is not found. It is different with the results obtained from the impact test of nanostructured Ti [23], in which the impact energy also increases at cryogenic temperature. The associated reason is as follows. As the temperature decreases, the energy obtained by the atom is small, and the resistance of the dislocation motion is increased, that is, dislocation pile-up. The elastic energy of the front part of the dislocation pile-up group is difficult to conduct and release through the dislocation motion in the adjacent crystal grains. When the energy of the accumulation is large enough, micro-cracks are formed on the dissociation crystal plane and extends along the cleavage plane and through the grain interior or grain boundary, causing the sample to break, thereby a decrease in the toughness of the material. When the temperature is higher, the dislocation plug-up group of adjacent dislocation sources can gain energy and move, resulting in the release of the accumulated energy without causing dissociation fracture [18] [22]. At the same time, the number of active dislocations is large, which leads to the number of voids gathered around the inclusions is large. As the voids gather and grow, the dimple fracture trend is becoming obvious. In addition, when the temperature decreases, the deformation mechanism changes from dislocation slip to twinning. Thus the impact toughness of Ti-6Al-4V titanium alloy at low temperature is higher
than that of many cold-short materials. It is probably due to the low content of self-interstitial atoms [24]. The interstitial atom is a kind of point defect, which causes lattice distortion. The lattice distortion suppresses the twinning at low temperatures [13] [25]. The Ti-6Al-4V titanium alloy is a kind of low-interstitial-element titanium alloy, thus it can maintain good toughness at cryogenic temperature. Furthermore, as is known to us that the plasticity and toughness of bcc β phase decrease with the decrease of temperature. Ti-6Al-4V titanium alloy contains less β phase, which allows them to maintain a certain toughness at low temperatures.

4. Conclusions
This work investigated the influence of varying temperatures (20°C~ -196°C) on the impact properties of Ti-6Al-4V titanium alloy, i.e. impact energy and fracture morphology. Based on the experimental results, the major conclusions are summarized as follows:

1. The impact absorbing energy and impact toughness of Ti-6Al-4V titanium alloy decrease with the decrease of temperature under the given range of 20°C to -196°C, wherein the impact energy at -196°C drops faster than that at -160°C. Meanwhile, the impact toughness of Ti-6Al-4V titanium alloy shows the same varying trend as the impact absorbing energy.

2. With the decrease of temperature, the brittleness of Ti-6Al-4V titanium alloy increases, but the plastic deformation zone featured with fiber area and shear lip area decreases as well as the impact toughness. However, the ductile fracture can be observed in the fracture morphology even at -196°C.

3. As the temperature decreases from 20°C to -160°C, the dimple size in the fracture zone and the cleavage planes decrease and increase, respectively. However, the size of dimples become bigger at -196°C. In addition, some secondary cracks, i.e. micro-cracks, were found to occur in the fracture zone at low temperatures.

4. The investigation on the impact properties of titanium alloy under low temperatures can provide theoretical guidance for its mechanism analysis of cryogenic machining.
5. Declarations

**Availability of data and materials**

The datasets supporting the conclusions of this article are included within the article.

**Competing interests**

The authors declare no competing financial interests.

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**Authors’ Contributions**

Wei Zhao provided fundamental ideas and conducted proof reading of this paper. Wenjia Su was in charge of the trial and wrote the first draft. Liang Li made some critical revisions, and provided financial support. Ding Fang and Ni Chen assisted the trial and analysis. All authors read and approved the final manuscript.

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Figures

![Figure 1](image-url)

Figure 1

Metallographic structure of the tested material.
Figure 2
Schematic diagram of impact test.

Figure 3
Impact tester (a) and specimen clamping (b).
Figure 4
Schematic diagram (a) and photo (b) of ductile fracture morphology.

Figure 5
Relationship between impact energy and specimen temperature.
Macroscopic morphology of the fracture zone at 20°C (a), 0°C (b), -40°C (c), -80°C (d), -120°C (e), -160°C (f), and -196°C (g).
Percentage of brittle fracture depending on the temperature.

Figure 7
Figure 8

Crack direction of the impact specimen at 20°C (a), -40°C (b), -120°C (c), and -196°C (d).
Figure 9

Microscopic morphology of the fracture zone at 20°C (a), 0°C (b), -40°C (c), -80°C (d),
-120°C (e), -160°C (f), and -196°C (g).