EFFECTS OF CRYOGENIC TREATMENT ON MICROSTRUCTURE AND WEAR RESISTANCE OF Fe-0.35C-6.3Cr MARTENSITIC STEEL

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Abstract

The study is conducted to determine the effect of quenching and tempering processes on microstructural evolutions and abrasive properties of medium carbon-high chromium steel. For this purpose, Austenitizing was performed at the temperatures of 1000 °C for 15 min followed by oil quenching. To determine the optimum tempering temperature, tempering temperatures were selected in the temperature ranges of 350-550°C. The samples cryogenically treated immediately after quenching in liquid nitrogen (-196°C) for 24 hrs. Dry sand/rubber wheel test was used to evaluate the wear resistance properties. Microstructural observation, fractography and retain austenite was evaluated by optical and scanning electron microscopy and X-ray diffraction analysis. The results show that the best wear resistance can be obtained at the tempering temperature of 500°C, due to the reduction of a tendency to micro-cracking, decrease in internal stresses and improvement of impact energy. Observation of the worn surfaces revealed that the wear mechanisms after tempering at 450°C are a combination of abrasive, adhesive and fatigue wear. However, abrasive wear is the only active wear mechanism for specimen tempered at 500°C. In addition, wear resistance of deep cryogenically treated sample was significantly increased (about 25%) by the removal of retained austenite and formation of uniform fine carbides distribution in the matrix.

Keywords: Heat treatment, Deep cryogenic, Microstructure, Wear-resistant, Martensitic steel

1 Introduction

High-chromium steels are extensively used as an inner and outer diaphragm in the cement industry due to excellent wear resistant properties. However, over the occurrence of wear, the grooves become wider resulted in a lower life of work-piece [1]. In this regards, carbon plays an important role to achieve high hardness and wear resistance. In addition, chromium increases

hardenability and thereby reduces the critical cooling rate in order to form martensitic microstructure as well as precipitation of the secondary carbides [2].

In such steels, retained austenite is generally formed after quenching to ambient temperature. Cryogenic heat treatment immediately after quenching is a useful method to reduce retained austenite and to achieve highest wear resistance. Transformation of retained austenite to martensite and precipitation of very fine carbides are the main factors affecting the mechanical properties after cryogenic treatment [3].

Collins et al. [4] believe that the highly dense crystalline defects that occur during cooling down to below zero are the suitable sites for nucleation of very fine carbides in the next tempering treatment. Huang et al. [5] believe that deep cryogenic treatment not only leads to the formation of very fine carbides and increases the volume fraction of carbides, but also creates a very uniform distribution of carbides in the matrix. In another study by Akhbari Zadeh et al. [6] on steel D6, hardness was increased in deep cryogenic treatment by 3.2% compared to conventional heat treatment, and the wear behaviour showed a 40% improvement compared to non-cryogenic treatment. Amini et al. [7], the suggested that increase in the percentage of carbide and appropriate distribution of carbides in cryogenic treatment was demonstrated in comparison with conventional heat treatment. In addition, nanometer-sized carbide precipitates were observed in cryogenic treatment.

According to the author's knowledge, there has been a lack of systematic data and knowledge concerning the effect of quenching and tempering processes as well as cryogenic treatment on the microstructural evolutions and abrasive properties of Fe-0.35C-6.3Cr martensitic steel. Therefore, the aim of the present paper is to identify the optimum heat treatment condition for achieving the desired microstructure and mechanical properties.

2 Experimental procedure

The chemical composition of wear resistant martensitic steel is listed in Table 1.

Elements	С	Si	Mn	Р	S	Cr	Мо	Ni	Fe
wt%	0.35	0.5	0.8	>0.009	>0.005	6.3	0.07	0.15	Ball

Table 1 The chemical composition of wear resistant martensitic steel (wt %)

Dilatometer test was performed by Bahr 805A/D in order to determine the critical temperatures including AC₁, AC₃, Ms and M_f for choosing the proper austenitizing temperature and quenching media. The dilatometer samples were prepared according to the SEP1681 standard in the form of a cylinder with a diameter of 4 mm and a length of 10 mm, and they were heat-treated in a vacuum of 5×10^{-4} . For conventional heat treatment, all samples were austenitized at 1000°C for 15 minutes; and after being quenched in the oil environment, they were tempered at the temperature range of 550-350°C with intervals of 50°C for one hour (QT). In order to investigate the effect of deep cryogenic treatment after austenitizing at 1000°C and oil quenching, the specimens were immediately transferred into the liquid nitrogen (-196°C) and held for 24 hrs. The tempering process was conducted in the temperature range of 350-550°C with an interval of 50°C for an hour (DCT). The details of QT and DCT are illustrated in **Fig. 1**.

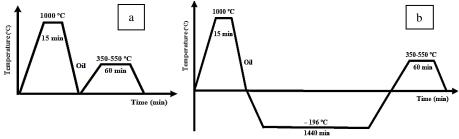


Fig. 1 Schematic of the heat treatment cycle a) QT and b) DCT

According to the ASTM E975-00 standard, X-Ray Diffraction spectroscopy (Philips PW3040) with Cuk_{α} radiator (λ =1.5405°A) was used. Amount of retained austenite in the structure can be determined by equation (1).

$$V_{\gamma} = \frac{1.4 I_{\gamma}}{I_{\alpha} + 1.4 I_{\gamma}} \tag{1.}$$

Where, V_{γ} is the volume fraction of austenite, and I_{γ} and I_{α} are X-ray diffraction intensity of planes (111) γ and (110) α , respectively [8].

Observations of microstructure and wear mechanisms were assessed by the aid of optical microscopy (Olympus, PME3) and scanning electron microscopy (Philips XL30). Villella solution (a mixture 1 gr acid picric + 10ml hydrochloric acid + 100 ml ethanol) was used as an etchant. Hardness value was measured by using a Rockwell test machine (Universal Coopa UVI) according to the ASTM E18 standard. The wear test was performed according to the ASTM G65–00 standards using a dry sand-rubber wheel device. In this device, the samples are placed against a rotating steel wheel with a rubber thread. The schematic of the wear testing device is shown in **Fig. 2**.

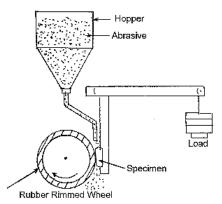


Fig. 2 Schematic of the dry sand-rubber wheel wear the testing device (ASTM G65–00)

The rubber wheel rotates at a constant speed of 200 rpm, and by applying pressure to the wheel/sample contact point, according to the type of method used, a vertical force of 130 N is applied. The abrasive material is silica sand (quartz sand) with the particle size of 212–300 μ m with less than 5 % moisture.

3 Results and discussion

3.1 Microstructure and microstructural transformations

In the dilatometry tests, the length and temperature variations of the specimens were printed during cooling and heating processes simultaneously in order to study phase transformations. According to **Fig. 3**, after heating the specimen, the length of the specimen increases due to thermal expansion; however, when a phase transformation of α ⁺ cementite to α + γ occurs, the specimen length decreases. The start and finish temperatures of this transition are 802°C (AC₁) and 861°C (AC₃), respectively. By increasing the temperature, the specimen length increases again. Holding the specimen in 1000°C does not cause its length to change. Upon quenching, the length starts to decrease due to thermal contraction. When the martensitic transformation begins at 238°C, the length suddenly increases at the temperature called MS. During cooling, the length increases continuously until the transformation finishes (M_f) at -1°C. So, the M_s and M_f temperatures of the steel were 238°C and -1°C, respectively. If the transformation of this steel had been interrupted by holding at some temperatures between M_s and M_f, martensite transformation might not have occurred. However, a certain percentage of soft austenite is retained when the specimen is quenched to room temperature. As a result, to decrease or eliminate the amount of retained austenite, the cryogenic treatment is necessary.

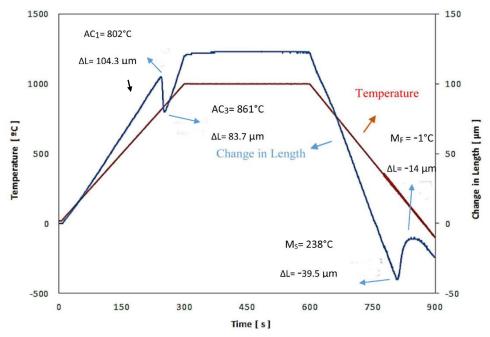


Fig. 3 Diagram of dilatometer test for determining critical temperatures

Fig. 4 shows the X-ray diffraction pattern of the quenched and cryogenic quenched samples. Calculating the percentage of retained austenite by using equation (1) indicates the presence of about 12% retained austenite in the quenched state and less than 1% after cryogenic treatment. **Fig. 4** also shows that the intensity of the austenite peaks decreases by performing a cryogenic treatment. Other researchers have proved the effect of cryogenic treatment on the reduction of retained austenite for other steels [9-11].

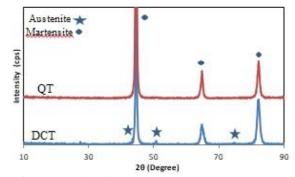


Fig. 4 XRD patterns after quenching, following conventional and cryogenic heat-treatment

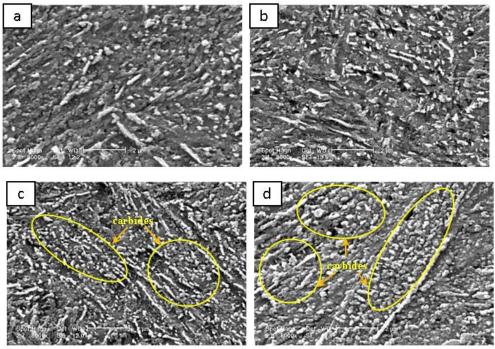


Fig. 5 SEM images of a and b) the quenched and tempered samples at 450 and 500°C, c) and d) the quenched –cryogenic and tempered samples at 450 and 500°C

Fig. 5 shows SEM images of quenched and tempered and quenched-cryogenic-tempered samples at the tempering temperatures of 450°C and 500°C. As shown in the images, in cryogenic treatments, the carbides are finer and more appropriately-distributed in the matrix. Increasing the percentage of carbides, finer carbides, and more appropriate distribution of carbides due to cryogenic treatment are the main factors in improving hardness and wear resistance in the cryogenically treated samples, which have also been confirmed by other researchers [4, 12]. Due to the cryogenic treatment, two mechanisms are involved: 1. transformation of retained austenite to martensite; 2. precipitation of very fine carbides. The reason for the precipitation of these carbides is that during cryogenic treatment, due to internal

microscopic stresses (caused by factors such as differences in thermal contraction coefficient of different phases and transformation of retained austenite to martensite), crystal defects such as twins and dislocations appear. Under this condition, if there is enough time, the regional penetration will lead to the clustering of carbon and alloying elements near these defects. On the other hand, supersaturation increases in martensite with a decrease in temperature; therefore, the lattice distortion and thermodynamic instability of the martensite increase. Both mentioned factors are the driving forces for the movement of carbon atoms and alloying elements around the crystalline defects resulting in very fine cores. These cores in tempering after cryogenic treatment lead to the formation of very fine (nanometer-sized) carbides. The increase in volume fraction of these deposits is indicative of local penetration of carbon atoms [5-6, 12-13].

Huang et al. [5] studied M2 high speed steel and showed that in deep cryogenic treatment, the volume fraction of carbides is doubled and a better distribution of carbides is provided. By increasing the tempering temperature from 450 to 500°C, the carbides grow and begin to diminish in number in both the conventional and the cryogenic treatment.

3.2 Investigation of mechanical properties

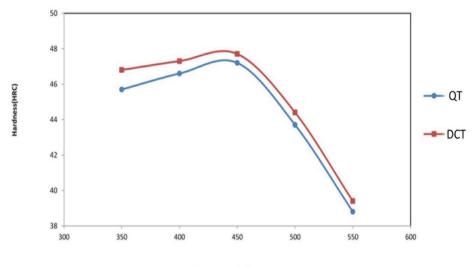
3.2.1 Hardness test

Variations of hardness with the tempering temperature of conventional and cryogenic treatments are shown in **Fig. 6**. As can be observed, by increasing the tempering temperature from 350 to 450° C, hardness increases due to the secondary hardness phenomenon caused by the precipitation of the alloyed M₇C₃ carbides and transformation of retained austenite to martensite [14, 15]. This increase in hardness is due to high hardness of this type of carbide and the integration between the network of the carbide particles and the martensitic matrix [15, 16]. Then, at the temperature range of 500-600°C, the hardness decreases because of the transformation of a large volume fraction of non-tempered martensite to tempered martensite, the transformation of the alloyed M₇C₃ carbides to M₂₃C₆, and the coarsening of carbides [14, 15].

In the study conducted by Balan et al. [17] on the martensitic stainless steel 16Cr-2Ni-0.15C, secondary hardness phenomenon at the temperature range of 350-450°C was observed. These researchers believed that the reason for this phenomenon (secondary hardness) is precipitation of the M7C3 carbides. They also considered the transformation of M7C3 carbides to M23C6 carbides and the coarsening of structures as the reasons for hardness decrease at the temperature range of 450-650°C. Also, in the study by Nasery et al. [14] on AISI 420 martensitic stainless steel, secondary hardness phenomenon at the temperature range of 400-500°C was considered to be due to the precipitation of the M7C3 carbide.

Increase in hardness with cryogenic treatment, as compared to conventional heat treatment, is related to complete transformation of retained austenite to martensite, precipitation of very fine carbides, and increase in the volume fraction of these carbides. Increase in hardness with cryogenic treatment has also been proved by other researchers [9-11]. In addition, in the cryogenically-treated samples, the increase in hardness at the temperature range of 350 to 450°C (secondary hardness) is less, compared to conventional heat treatment, due to the reduction in the retained austenite in the cryogenically-treated samples.

In the study carried out on the D2 steel by Das et al. [18], the increase in hardness in deep cryogenic treatment was 4.2% compared to conventional heat treatment. It shows that deep cryogenic treatment increases the hardness. This increase can be explained by retained austenite decreasing and higher content of secondary carbides.



Temperature (°C)

Fig. 6 Variations of hardness with the tempering temperature of conventional heat treatment and cryogenic treatment

3.2.2 The wear test

The wear test was performed using dry sand-rubber wheel wear testing device. **Fig. 7a** shows the weight loss of the samples at different tempering temperatures after 30 minutes in both conventional heat treatment and cryogenic treatment. As can be seen, in the conventionally and cryogenically-heat treated samples, the weight loss rate has decreased by increasing the tempering temperature from 350 to 500°C, the wear properties have improved, and the weight loss rate has increased at 550°C again.

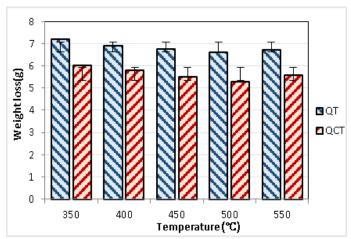


Fig. 7 a) Weight loss rate of the samples by increasing the tempering temperature in the conventionally and cryogenically-heat treated samples

At the temperature ranges of 350 to 450°C, by increasing the hardness of the samples, wear properties have also improved. At 500°C, due to hardness decrease, wear properties have

improved due to the reduction in internal stresses and increase in toughness of the matrix and consequently, reduction in the tendency to micro-crack. At 550°C, with respect to the increase in toughness of the matrix, wear properties have deteriorated, which is due to a significant reduction in hardness [19].

Gupta et al. [20] found that by increasing toughness up to a certain level, the wear resistance caused by abrasive particles increases, but further increase in toughness reduces the wear resistance, and after that, wear resistance depends on the hardness.

In the study on wear resistant low-alloy chrome-molybdenum steel Fe-0.6C-1.5Cr-0.4Mo conducted by Hangung et al. [21], it was found that wear resistance is related to hardness and toughness, and wear resistance has increased at 350°C where hardness and toughness are optimal. These results are consistent with those obtained by Xiang et al. [22] on the wear resistant steel Fe-0.75C-1Cr-0.8Mo.

According to the obtained results, the wear rate (weight loss) in the case of the cryogenicallyheat treated has decreased up to 16% to 20%, compared to the conventional heat treatment (Table2). In deep cryogenic treatment, reduction of internal stresses due to finer carbides leads to the reduction of a tendency towards micro cracking. His phenomenon, together with the more appropriate distribution of carbides and removal of retained austenite result in the increase in the wear resistance.

In other words, the main reason for the increase in wear resistance, in addition to the removal of retained austenite as a soft phase, is an increase in the percentage of the alloyed carbides due to cryogenic treatment and more uniform and homogeneous distribution of the carbides. Precipitation of fine carbides also reduces the percentage of carbon and alloying elements in the matrix; therefore, the toughness of the matrix phase increases. Precipitation of fine hard carbides and increase in the toughness of the matrix reduce the wear rate in the cryogenically-heat treated samples [5, 23-24].

In a study on 1.2436 DIN steel carried out by Akhbarizadeh et al. [6], in the case of deep cryogenic treatment, the wear behavior showed 40% improvement, compared to the non-deep cryogenic treatment. This is mainly attributed to the increase in the carbide particles and their uniform distribution in that matrix. In another study on the 80CrMo125 tool steel conducted by Amini et al. [9], the reason for the 37% to 52% increase in wear resistance in deep cryogenic treatment, compared to conventional heat treatment, is mentioned to be the precipitation of very fine carbides and complete transformation of retained austenite to martensite. In order to determine the wear mechanism, the surfaces of samples were analyzed by scanning electron microscopy. **Fig. 8** shows the wear surfaces of the samples tempered at 450 and 500°C. In this figure, a series of scratches and pitting are observed. The parallel wears created on two surfaces are the result of micro-ploughing abrasive wear mechanism [25,26]. The particles sticking to the surface, which are in fact the result of wear, indicate the adhesive wear mechanism [27-30]. By increasing the tempering temperature, the pitting on the surface is reduced.

Fig. 9 shows the SEM images of the wear surface in the quenched-deep cryogenic treated and tempered conditions at 450 and 500°C. As can be seen in this figure, some parallel scratches have also been created on the surface because of the micro-cutting abrasive wear. With an increase in the tempering temperature from 450 to 500°C, the width and the depth of the grooves (formed on the surface due to the decreased hardness and increased toughness of the matrix) get smaller. By making a comparison between the surfaces of conventional heat treatment and cryogenic treatment (**Fig. 8** and **Fig. 9**), it is observed that at the same tempering temperature, the amount of created scratches and pitting has decreased. Moreover, the adhesive wear

mechanism in cryogenic treatment has been removed, and these factors result in the improvement of the wear properties in cryogenic treatment. The removal of the adhesive wear mechanism in the cryogenically-treated samples is related to the hardness increase in this sample. In the studies on tool steels conducted by Fantalvo [31], increasing the volume fraction and reducing the distance between carbides have reduced the adhesive wear. On the other hand, Yang et al. [32] have considered increasing the hardness of the steel surface as the main actions of the resistance to adhesive wear. Therefore, resistance to adhesive wear increases in the deep cryogenic treatment because of the increase in hardness resulting from an increase in the volume fraction of carbides. Considering the above-mentioned points, in the deep cryogenically-treated samples, due to the increase in volume fraction of the carbides and their getting finer and the subsequent increase in hardness (because of the increased percentage of martensite and fine carbide precipitations), the adhesive wear is reduced. In addition, as the carbides get finer, the internal stresses are reduced and; hence, the tendency to form fine cracks decreases, which results in the improvement of wear resistance [33].

At the tempering temperature of 450°C of cryogenic treatment and conventional treatment, pitting and cavities can be observed on some parts of the surface. In order to better investigation of wear mechanism, samples were cross sectioned.

 Table 2 Improvement in wear behavior in DCT samples compared QT samples at different tempering temperatures

(°C) Temperature	The percentage of improvement in wear behavior (weight loss) in DCT samples compared to QT samples
350	16.53
400	16.84
450	18.64
500	20.24
550	17.11

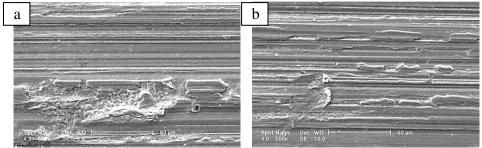


Fig. 8 SEM images of the wear surface of the quenched and tempered samples at, a) the tempering temperature of 450°C, b) the tempering temperature of 500°C

Fig. 10 shows the SEM images of the surface perpendicular to the direction of the quenched and tempered samples at 450 and 500°C. At 450°C, in addition to asperities caused by micro-cutting, a series of pitting and cracks are created under the surface that has been formed due to the mechanism of micro-crack and fatigue (**Fig. 10, a**). By increasing the tempering temperature to 500°C, just asperities of micro-cutting are seen; and because of the reduction in hardness and increase in the toughness of the sample, there is no mechanism of micro-crack and fatigue.

Therefore, the wear properties of the tempered sample at 500°C have improved compared to the tempered sample at 450°C (**Fig. 10, b**) [29].

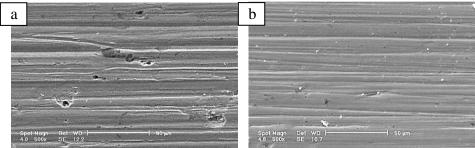


Fig. 9 SEM images of the wear surface of the quenched-deep cryogenically- treated and tempered samples at, a) the tempering temperature of 450°C, b) the tempering temperature of 500°C

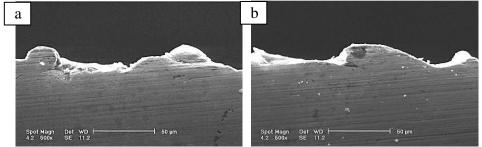


Fig. 10 SEM images of the wear cross-section of the quenched and tempered samples at, a) the tempering temperature of 450°C, b) the tempering temperature of 500°C

Fig. 11 shows the SEM images from the cross section of the quenched-deep cryogenicallytreated and tempered samples at 450 and 500°C. Investigation of these images shows a similar behavior of the samples with conventional heat treatment.

A comparison of **Figs. 10** and **11** reveals that at the identical tempering temperature, the depth of the grooves caused by micro cutting has decreased in cryogenic treatment, compared to conventional treatment. This is justifiable with regard to the improvement in hardness and wear properties of the cryogenically-treated samples, compared to the conventionally-heat treated samples.

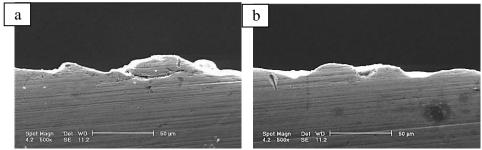


Fig. 11 SEM images of the wear cross-section of the quenched-deep cryogenically-treated and tempered samples at, c) 450°C, d) 500°C

4 Conclusion

- Based on the dilatometer results, the finishing temperatures of martensitic transformation is (MF) -1°C and there will be some retained austenite in the samples at room temperature; therefore, it is necessary to use cryogenic treatment for the removal of retained austenite.
- 2) As a result of cryogenic treatment, compared to the conventional heat treatment, hardness is improved. The reason is related to the elimination of retained austenite, carbide precipitation, and more appropriate distribution of the carbides.
- 3) The secondary hardness of this steel was observed at a temperature range of 350 to 450° C, which is due to the creation of the alloyed M_7C_3 carbides. At the temperature range of $500-550^{\circ}$ C, the hardness decreases due to the transformation of a significant volume fraction of the non-tempered martensite into tempered martensite, the transformation of the alloyed M_7C_3 carbides to $M_{23}C_6$, and the coarsening of the carbides.
- 4) At the temperature range of 350 to 450°C, as the hardness of the samples increases, the wear properties also improve. However, at 500°C, with respect to the reduction in hardness, the wear properties have improved, which is attributed to the reduction in internal stresses, the increase in the matrix toughness, and reduction in micro-cracking tendency. At 550°C, with respect to the increase in the matrix toughness and significant decrease in hardness, the wear properties have declined.
- 5) The cryogenic treatment, as compared to conventional heat treatment, has caused 17% to 20% enhancement in the wear properties. This is related to the elimination of retained austenite, precipitation of fine carbides, and better distribution of the carbides.
- 6) The wear mechanism in the conventionally-heat treated samples at 450°C is adhesive wear, fatigue, micro-cutting scratches, and micro-cracking scratches; and at the tempering temperature of 500°C, it is adhesive wear mechanism and fine cut.
- 7) The wear mechanism in the cryogenically-treated samples at the tempering temperature of 450°C is micro-cutting abrasive wear, micro-cracking scratches and fatigue; and at the tempering temperature of 500°C, the mechanism is micro-cutting scratches.
- 8) In the cryogenically-treated samples, the hardness increases due to the increase in the percentage of carbides and their appropriate distribution and the removal of retained austenite; thus, increase in hardness leads to the removal of the adhesive wear mechanism and, as a consequence, the wear properties improve.

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