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Abstract

Continuous cooling transformation (CCT) characteristics of microalloyed steels with different tungsten (W) contents (0, 0.1 and 1. wt.%) were investigated to obtain the necessary information for heat treatment of these steels. The effects of W addition on the sizes of prior austenite grains and precipitates were analysed. CCT diagrams were obtained by varying the cooling rates from 0.1 to 120. °C/s. Transformation characteristics were determined by using dilatometer test, microscopic observation and hardness measurement. The results showed that W had a positive effect on the refinement of prior austenite grains and precipitates. The CCT diagrams exhibited that the ranges of transformation products were shifted to the right side of the diagram when the W content increased. CCT diagram for steel with 0.1% W was similar in shape to that without W. The addition of 1% W induced two separated transformation ranges in the cooling rate range of 0.1 to 1. °C/s in the diagram. Both the austenitisation starting and finishing temperatures were raised as W was added. W addition induced decreased critical cooling rates for phase transformations and obtaining complete ferrite + pearlite microstructures. The martensite transformation temperature was decreased after W addition. The addition of W caused increased hardness, and the hardness obeyed an exponential type relationship with cooling rate.

Keywords

characteristics, transformation, effects, tungsten, steels, continuous, microalloyed, cooling

Disciplines

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Effects of tungsten on continuous cooling transformation characteristics of microalloyed steels

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Abstract

Continuous cooling transformation (CCT) characteristics of microalloyed steels with different tungsten (W) contents (0, 0.1 and 1 wt.%) were investigated to obtain the necessary information for heat treatment of these steels. The effects of W addition on the sizes of prior austenite grains and precipitates were analysed. CCT diagrams were obtained by varying the cooling rates from 0.1 to 120°C/s. Transformation characteristics were determined by using dilatometer test, microscopic observation and hardness measurement. The results showed that W had a positive effect on the refinement of prior austenite grains and precipitates. The CCT diagrams exhibited that the ranges of transformation products were shifted to the right side of the diagram when the W content increased. CCT diagram for steel with 0.1% W was similar in shape to that without W. The addition of 1% W induced two separated transformation ranges in the cooling rate range of 0.1 to 1°C/s in the diagram. Both the austenitisation starting and finishing temperatures were raised as W was added. W addition induced decreased critical cooling rates for phase transformations and obtaining complete ferrite+pearlite microstructures. The martensite transformation temperature was decreased after W addition. The addition of W caused increased hardness, and the hardness obeyed an exponential type relationship with cooling rate.

Keywords: Microalloyed steel; Tungsten; CCT diagram; Precipitate; Microstructure

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1. Introduction

Microalloyed steels are widely used in the fields of oil and gas extraction [1], construction [2] and transportation [3]. Several aspects of microalloyed steels, including heat treatment [4], phase transformation [5,6], mechanical properties [7,8] and oxidation behaviours [9], have been studied over the past decades. Multiple microstructures in microalloyed steels obtained depend on the states of the austenite prior to the transformations, chemical compositions and cooling conditions. Different microstructural combinations bring about drastic change in the mechanical properties. Austenite is the parent phase of all the microstructures including pearlite, proeutectoid phases, bainite, martensite and various ferritic microstructures [10]. Depending on chemical composition and cooling rate, the austenite of a given microalloyed steel could transform to all of the listed microstructures. The microstructures of austenite transformation products depend on austenite grain size. Refinement of austenite grain size in microalloyed steels is critical in producing the final refined microstructures with high strength and toughness. By adjusting the chemical compositions, the mechanical properties of microalloyed steels can be significantly tailored. Previous studies have been carried out about the effects alloying elements on the microstructures and mechanical properties of these steels. Balart et al. [11] studied the effect of Si and Ti on the microstructure and mechanical properties of V-microalloyed steel. They found that Si and Ti additions show positive effect in increasing the strength and refining the austenite grain size, respectively. As reported by Najafi et al. [12], the presence of microalloying elements significantly enhanced the strength of as-cast microalloyed steels due to the formation of fine-scale precipitates. The work of Han et al. [13] indicated that precipitates affect the hot ductility of microalloyed steels.

Cooling procedure is one of the most important processing factors affecting the mechanical properties of microalloyed steels after heat treatment or hot working. A fast cooling rate depresses the transformation

temperature and refines precipitate size, and hence increases the hardness of microalloyed steels [14]. The microstructure is changed from ferrite/pearlite to bainitic ferrite with an increase of cooling rate, and such microstructural change is responsible for the high strength-toughness combination of microalloyed steels at high cooling rate [15]. The work of Rasouli indicated that the best combination of strength and ductility of microalloyed forging steel can be obtained by adjusting the microstructural component through control of the cooling rate [16].

Tungsten (W) is a strong ferrite former, and is effective for precipitate refining and solid solution strengthening. As the content of W increases in alloy steels, W forms very hard, abrasion-resistant carbides, which can prevent grain growth at high temperature. In microalloyed steels, the microstructures and mechanical properties are greatly dependent on the W contents [17,18]. The transformation characteristics in W-containing microalloyed steels under different cooling conditions become more complicated relative to those of W-free microalloyed steels due to the significant effect of W on the microstructural evolution. For obtaining the best combination of excellent strength and toughness which are closely related to the microstructures, it is essential to investigate the transformation characteristics at various cooling rates to obtain the necessary information for heat treatment and hot working of these steels. Unfortunately, the effects of W on the microstructural evolution of microalloyed steels at different cooling rates have not been well reported, and no detailed mechanism for these is presented.

A review of the effects of some alloying elements (including W, Cr, V and Mo etc.) on the transformation diagrams was presented in an earlier paper [19]. All these studies were conducted under isothermal conditions. For application to most practical processes, however, continuous cooling transformation (CCT) data are needed. The aim of this study is to systematically investigate the CCT characteristics of

microalloyed steels with different W contents. The effects of W addition on the prior austenite grain size will be investigated first, and CCT diagrams of microalloyed steels with different W contents will be presented. The hardness change and microstructural evolution with cooling rates will also be analysed. Particular discussion will be made on the dependence of microstructural combination on the W content and cooling rate.

2. Experimental procedure

Three kinds of microalloyed steels with different W contents (0, 0.1 and 1 wt.%) were used in this study. The chemical compositions of the steels are given in Table 1. Ingots were homogenised at 1230°C for 1 h and then hot forged to be a cube with section size of 140 mm × 140 mm and height of 400 mm followed by air cooling. The final forging temperature was controlled to be higher than 950°C. Cylindrical dilatometer specimens with a diameter of 3 mm and height of 10 mm were machined from the forged pieces.

Different phases, such as austenite and ferrite, in steels have distinctly different specific volumes. Hence, it is possible to differentiate the volume change (length change) when phase transformation occurs from the linear thermal expansions during heating and cooling of steels by employing a high resolution dilatometer. In the present study, dilatometer specimens were heated to the temperature of 950°C at a rate of 20°C/s, then held for 10 min, and finally cooled to room temperature at different linear cooling rates ranging from 0.1 to 120°C/s. The schedule for dilatometer tests is schematically shown in Fig. 1. Dilatometer tests were conducted on a Theta Dilatronic III dilatometer. The specimens were heated and cooled under a vacuum of 5×10^{-4} mbar to prevent oxidation.

During phase transformation, the specimen expands during cooling although the temperature decreases as a

result of the microstructure rearrangement. A typical dilatation vs. temperature curve of 1W steel at the cooling rate of 0.5°C/s is shown in Fig. 2. The up and down arrows indicate the progress of heating and cooling cycles, respectively. From this curve, the start and finish of a phase transformation can be easily detected. However, as the start of pearlite transformation cannot be clearly detected on the dilatation-temperature curve [20,21], metallographic observation is needed to determine the starting temperature of pearlite transformation. In this research, all the specimens after dilatometer tests were subjected to detailed microscopic observation and hardness measurements to ensure that every phase transformation temperature was accurately determined.

Metallographic specimens were etched with 2% nital solution for microstructural observation by an OLYMPUS BX51M optical microscope (OM). Hardness was measured with a Vickers hardness tester using 2 Kg load and 10 s dwelling time, and 6 indentations for every specimen were randomly made on its surface. For observing prior austenite grain boundaries, specimens were re-heated to 950°C at a rate of 20°C/s for 10 min followed by water quenching. The water quenched specimens were etched with a “picric acid + FeCl₃ + dodecyl benzene sulfonic acid sodium salt” solution to reveal prior austenite grain boundaries, and then observed by OM. To examine the characteristics and compositions of precipitates in the water quenched specimens, extraction replicas as well as thin foils mounted on Cu grids specimens were prepared and analysed using energy dispersive X-ray spectroscopy (EDS) method on JEOL JEM-2100F transmission electron microscope (TEM) operated at 200 kV.

3. Results

3.1. Prior austenite grains

Fig. 3a and b present the prior austenite grains of the water-quenched 0W and 1W steels after holding at 950°C/s for 10 min, respectively. It can be seen that the addition of 1% W induces finer austenite grains in contrast to that in 0W steel. The average austenite grain sizes of the 0W and 1W steels were determined as 15.8 and 10.7 μm , respectively, by using circular intercept method according to ASTM: E112-10. W addition shows a positive effect on decreasing the austenite grain size.

3.2. CCT diagrams

CCT diagrams serve an incredibly useful purpose in representing the transformation characteristics of steels which are not isothermally heat treated, and in revealing the role of alloying elements in affecting the microstructures of the steels. For studying the effects of W addition on non-isothermal transformations of microalloyed steels, CCT diagrams have been obtained for the steels with different W contents. Figs. 4 and 5 present the CCT diagrams of steels with and without W additions, respectively. In these diagrams, F is ferrite, P is pearlite, B is bainite, M is martensite, M_s is the starting temperature of martensite transformation and M_f is the finishing temperature of martensite transformation. Representative cooling curves, cooling rates, transformation ranges and the nature of the transformation products after continuous cooling are shown in the diagrams.

As shown in Fig. 4, the CCT diagram of 0W steel exhibits consecutive characteristic of the phase transformation and multi-transformation curves, revealing complex microstructures as cooling rate is increased from 0.1 to 120°C/s. Ferrite, pearlite, bainite and martensite are involved in the diagram. Ferrite forms throughout the cooling rate range from 0.1 to 120°C/s. Pearlite is produced under a cooling rate range from 0.1 to 20°C/s. Bainite begins to be produced by increasing the cooling rate higher than 5°C/s. Martensite

is observed at the cooling rate higher than 40°C/s, and the value of M_f shows a decreasing trend with an increase of cooling rate. M_f is dependent on the cooling rate and can therefore be varied by changing the cooling rate.

The shape of the CCT diagram for 0.1W steel is similar to that for 0W steel, as shown in Fig. 5a. The difference is that the ferrite, pearlite, bainite and martensite transformation ranges in 0.1W steel are shifted to a longer time side as compared with that in 0W steel. Moreover, the addition of W narrows the range of pearlite transformation. Significant effect of W on the CCT diagram is observed when its content is increased to 1%. As shown in Fig. 5b, in the cooling rate range of 0.1 to 1°C/s, the transformation ranges can be classified in the higher temperature range (above 625-640°C) and the lower temperature range (below 538-599°C). The two transformation ranges are separated by a no-transformation range in which no progress of the transformation is observed on cooling. Transformation products are ferrite and pearlite in the higher temperature range, and only bainite in the lower temperature range. Bainite transformation occurs even though at the very low cooling rate of 0.1°C/s. In addition, as the W content is increased to 1%, a further shift of the ranges of transformation products to the right side of the CCT diagram is caused in contrast to that for 0.1W steel, as shown in Fig. 5a and b.

The measured A_{c1} and A_{c3} temperatures for 0W, 0.1W and 1W steels are: $A_{c1}=766^\circ\text{C}$ and $A_{c3}=903^\circ\text{C}$ for 0 W steel; $A_{c1}=767^\circ\text{C}$ and $A_{c3}=906^\circ\text{C}$ for 0.1W steel; and $A_{c1}=771^\circ\text{C}$ and $A_{c3}=910^\circ\text{C}$ for 1W steel. Obviously, W addition induces increased A_{c1} and A_{c3} temperatures. The values of M_s are obtained to be 434, 429 and 420°C for 0W, 0.1W and 1W steels, respectively. W shows a positive effect on decreasing the starting temperature of martensite transformation. Fig. 6 shows the variation of the starting temperatures of austenite to ferrite (A_{f3}) as a function of cooling rates. It can be seen that the addition of W increases A_{f3} and shifts the A_{f3}

temperature to lower cooling rates.

The critical cooling rates for the start of transformations of ferrite, pearlite, bainite and martensite, and for obtaining complete ferrite+pearlite are summarised in Table 2. It can be seen that W addition induces decreased critical cooling rates. Ferrite and pearlite are more readily formed in 0W steel than that in W-containing steels. W promotes the formation of bainite and martensite even though at a very low cooling rate. Especially, when the W content is increased to 1%, bainite is formed at the lowest cooling rate of 0.1°C/s, and no complete ferrite+pearlite microstructure is formed in the cooling rate range of 0.1 to 120°C/s.

3.3. Hardness

The relationship between hardness and cooling rate is presented in Fig. 7. It can be seen that the addition of W brings about increase in the hardness at a given cooling rate. For 0W and 0.1W steels, the hardness shows a gradually increasing trend with an increase of cooling rate. For 1W steel, however, the hardness increases significantly in the cooling rate range of 0.1 to 40°C/s, then increases slightly when the cooling rate is higher than 40°C/s.

In order to describe the change of hardness of the tested cylindrical specimens (diameter is 3 mm, and height is 10 mm), an exponential equation proposed by Wang et al. [22] was introduced in this study:

$$HV=a+b*\exp(c*v)+d*\exp(e*v) \quad (1)$$

where HV is Vickers hardness (HV2), v is the cooling rate (°C/s), a , b , c , d and e are constants. The quantitative expressions between HV and v for 0W, 0.1W and 1W steels can be acquired through regression analysis, and the adjust coefficient of determination R^2_{adj} can be obtained to evaluate the performance of the

calculated equations. The calculated values of a , b , c , d and e , as well as relevant R^2_{adj} , are given in Table 3 for each steel condition. Fig. 8 presents the histograms of residual diagnosis results for the 0W, 0.1W and 1W steels after regression analysis. It can be seen that all the residuals are normally distributed; indicating accurate calculation results can be obtained [23]. Fig. 9 shows the comparative plots for the tested and calculated hardness values at different cooling rates. It can be seen that the results of calculation agree well with that of experiment.

3.4. Microstructural evolution

The microstructures of the continuously-cooled 0W, 0.1W and 1W steels are presented in Fig. 10. The label at the upright corner of each image indicates the cooling rate. The microstructure of 0W steel at the cooling rate of 0.5°C/s is composed of ferrite and pearlite (Fig. 10a). When the cooling rate is increased to 30°C/s, the microstructure becomes a mixture of ferrite and bainite (Fig. 10b). At the highest cooling rate of 120°C/s, the microstructure comprises martensite, bainite and small quantity of ferrite, and predominately martensite, as shown in Fig. 10c. After 0.1% W addition, the microstructure of 0.1W steel is still composed of ferrite and pearlite when the cooling rate is 0.5°C/s (Fig. 10d). As the cooling rate increases, the 0.1W steel exhibits a mixed microstructure of ferrite, bainite and martensite at the cooling rate of 30°C/s (Fig. 10e), and then a fully martensitic microstructure at the cooling rate of 120°C/s (Fig. 10f). 1% W addition shows significant effect on the microstructures after continuous cooling in contrast to the 0W and 0.1W steels. At the cooling rate of 0.5°C/s, bainite is observed in the microstructure of 1W steel in addition to ferrite and pearlite (Fig. 10g). As the cooling rate is increased to 30°C/s, the microstructure of 1W steel is mainly composed of martensite, and small quantity of bainite and ferrite (Fig. 10h). At the highest cooling rate of 120°C/s, 1W steel exhibits a full martensite microstructure, as shown in Fig. 10i. The microstructural observation obtained

from Fig. 10 reveals that W plays an important role in obtaining different microstructural combinations during continuous cooling process.

4. Discussion

In microalloyed steels, the austenite grain growth behaviour is closely related to the state of precipitates. Fine precipitates effectively inhibit austenite growth during reheating, and the more stable the precipitates, the more effectively grain growth is inhibited to higher temperatures [4,24]. W is a strong carbide-forming element. When added to steel, W forms hard and stable carbides or complex carbides with other carbide-forming elements, such as Ti, Nb and V. W shifts the eutectoid point towards lower carbon concentrations, and induces the precipitation of fine carbides evenly distributed in the steel matrix [25]. Fig. 11 shows the characteristics and compositions of precipitates in both 0W and 1W steels. It can be seen that precipitates have been significantly refined after 1% W addition relative to the steel without W, as shown in Fig. 11a and b. EDS analysis indicates that the particles in 0W steel contain Ti, Nb and V elements (Fig. 11c), and W exists in the particles in 1W steel (Fig. 11d). Based on the chemical compositions given in Table 1 and the previous work [4], these particles are probably complex Ti, Nb and V carbides/nitrides (carbonitrides). According to these results, it is believed that W addition induces refined precipitates, which subsequently retards the growth of austenite grains. This result is consistent with the fact that W has a positive effect on the refinement of austenite grains [18].

W produces both quantitative and qualitative changes in the kinetics of transformation characteristics. The results obtained by Totten et al. [26] revealed that W shifts the transformation ranges to the longer time side of the isothermal transformation diagrams, and two separated transformation ranges in the diagram will be caused as the steel is alloyed with high content of W, as shown in Fig. 12. This fact is consistent with the

results obtained from Figs. 5 and 6 that the transformation ranges are shifted to the right side of the CCT diagram after 0.1% W is added, and two transformation ranges are separated when the W content is increased to 1%.

As a strong ferrite former, W effectively stabilise ferrite by raising the ferrite $\rightarrow$ austenite temperature. The ferrite field will be expanded as W is added. As a result, the A_{c1} and A_{c3} temperatures during heating, and the A_{r3} temperature during cooling show increasing trends with W [27]. M_s and the factors that affect its value have been broadly investigated. M_s is a definite temperature for given steel. M_s strongly depends on the chemical compositions of the steel. Several studies have indicated that W depresses the M_s temperature [28,29]. Austenite grain size also plays an important role on affecting M_s . It is well accepted that refined austenite grain size induces decreased M_s [30,31]. Increase of W content results in smaller austenite grain size (Fig. 3), which in turn decreases the M_s temperature. It has been suggested that the effects of different alloying elements, such as W, on M_f are similar in magnitude to their effect on M_s [32,33], i.e. M_f will decrease when M_s is decreased.

The hardness of steels is strongly related to the chemical compositions and final microstructures. Improvement of hardness after W addition has been discussed in our previous work [18]. With an increase of the applied cooling rate, the transformed structure evolves from ferrite+pearlite/bainite (0W and 0.1W: ferrite+pearlite; 1W: ferrite+pearlite+bainite) to final martensite. The content of bainite and martensite in the final microstructure gradually increases as the cooling rate increases, as shown in Figs. 4, 5 and 10. Consequently, the hardness increases with an increase of the cooling rate due to the changes in the microstructural combination [34].

There is little existing experimental research on systematically studying the effects of W on the CCT characteristics of microalloyed steels. In this study, the CCT diagrams of microalloyed steels with different W contents have been created and presented. The phase transformation behaviour and the microstructural evolution at different cooling rates have been analysed in detail. The research has immediate practical applications, and the outcomes can be successfully applied in the heat treatment and hot working of these steels in steel manufacturing industries.

5. Conclusions

CCT diagrams for microalloyed steels with different W contents were determined, and the transformation characteristics during continuous cooling were investigated. The dependence of microstructural combination on the W content and cooling rate was analysed. Following conclusions are drawn from the present work:

- (1) The prior austenite gains and precipitates are refined after W addition. The ranges of transformation products are shifted to the right side of the diagram as the W content is increased.
- (2) The shape of the CCT diagram for steel 0.1W is similar to that for steel 0W. After 1% W addition, two transformation ranges are separated by a no-transformation range in the cooling rate range of 0.1 to 1°C/s in the diagram.
- (3) Both the austenitisation starting and finishing temperatures during heating are raised after W addition. The critical cooling rates for phase transformations and obtaining complete ferrite+pearlite microstructures decrease as the W content is increased.
- (4) W addition induces decreased martensite transformation temperature and increased hardness. The quantitative relationship between hardness and cooling rate can be expressed by an exponential type equation.

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Figure Captions:

Fig. 1. Schematic illustration of the schedule for dilatometer tests.

Fig. 2. Dilatation vs. temperature curve of 1W specimen at the cooling rate of 0.5°C/s. Ac_1 and Ac_3 are austenitisation starting and finishing temperatures, respectively, during heating; I=Austenite→Ferrite→Pearlite; II=No Transformation; and III= Austenite:Ferrite:Pearlite→Bainite.

Fig. 3. Prior austenite grains of (a) 0W and (b) 1W steels after holding at 950°C/s for 10 min.

Fig. 4. CCT diagram of 0W steel.

Fig. 5. CCT diagrams of W-containing steels. (a) 0.1W and (b) 1W

Fig. 6. Dependence of A_{r3} on cooling rate of the studied steels.

Fig. 7. Dependence of hardness on cooling rate of the studied steels.

Fig. 8. Histogram of residual diagnosis results for the (a) 0W, (b) 0.1W and (c) 1W steels after regression analysis.

Fig. 9. Comparison between the tested and calculated hardness values at different cooling rates.

Fig. 10. Microstructures of continuously-cooled specimens. (a,b,c) 0W, (d,e,f) 0.1W and (g,h,i) 1W.

Fig. 11. Characteristics and compositions of precipitates in (a,c) 0W and (b,d) 1W steels. (a,b) Bright field images and (c,d) EDS spectra.

Fig. 12. Isothermal transformation diagrams of carbon steel and steel alloyed with W [26].

Table Captions:

Table 1 Chemical compositions of the investigated steels (wt.%).

Table 2 Critical cooling rates for phase transformations and obtaining complete ferrite+pearlite.

Table 3 Calculated parameters for the exponential equation.

Fig. 1. Schematic illustration of the schedule for dilatometer tests.

Fig. 2. Dilatation vs. temperature curve of 1W specimen at the cooling rate of 0.5°C/s. Ac_1 and Ac_3 are austenitisation starting and finishing temperatures, respectively, during heating; I=Austenite→Ferrite→Pearlite; II=No Transformation; and III= Austenite:Ferrite:Pearlite→Bainite.

Fig. 3. Prior austenite grains of (a) 0W and (b) 1W steels after holding at 950°C for 10 min.

Fig. 4. CCT diagram of 0W steel.

(a)

(b)

Fig. 5. CCT diagrams of W-containing steels. (a) 0.1W and (b) 1W.

Fig. 6. Dependence of A_{r3} on cooling rate of the studied steels.

Fig. 7. Dependence of hardness on cooling rate of the studied steels.

(a)

(b)

(c)

Fig. 8. Histogram of residual diagnosis results for the (a) 0W, (b) 0.1W and (c) 1W steels after regression analysis.

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Table 1 Chemical compositions of the investigated steels (wt.%).

Steels	C	Mn	Cr	Ni	Cu	Si	Al	V	Nb	Ti	P	S	N	W
0W	0.171	1.22	0.103	0.025	0.023	0.469	0.019	0.015	0.023	0.019	0.013	0.008	0.007	0
0.1W	0.170	1.20	0.103	0.024	0.024	0.471	0.024	0.015	0.023	0.020	0.014	0.009	0.008	0.09
1W	0.171	1.20	0.112	0.024	0.022	0.499	0.021	0.016	0.020	0.020	0.008	0.008	0.006	0.99

Table 2 Critical cooling rates for phase transformations and obtaining complete ferrite+pearlite.

Steels	F starting	P starting	B starting	M starting	Complete F+P
0W	-	20°C/s	5°C/s	40°C/s	2°C/s
0.1W	60°C/s	10°C/s	2°C/s	30°C/s	1°C/s
1W	30°C/s	2°C/s	-	20°C/s	-

Table 3 Calculated parameters for the exponential equation.

Steels	a	b	c	d	e	R^2_{adj}
0W	454.949	-50.603	-0.019	-248.383	-0.019	0.994
0.1W	453.420	-47.542	-0.025	-244.496	-0.025	0.992
1W	449.360	-241.528	-0.051	-1073.944	-33.101	0.999