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AN INVESTIGATION OF OVER-HEATING AND ROCK-LIKE FRACTURE OF STEEL 18Cr2Ni4WA

By

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An Investigation of Over-heating and Rock-like Fracture of Steel 18Cr2Ni4WA

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Abstract

We compared the sensibility to over-heating and examined the rock-like fracture of five sets of steel 18Cr2Ni4WA produced by three different processes (a mixture of open hearth and electric processes through RH vacuum circulating deoxidization; basic electric process; and electric process plus electric slag remelting) and two different methods of deoxidization (using aluminum and titanium or aluminum alone). Experiments indicate that the conditions that cause this kind of steel to have serious over-heating and rock-like fracture are: (1) when the steel is heating at high temperature, primary austenite grains are in full growth, (2) the steel contains MnS inclusion which in the form of small grains precipitates at austenite grain boundary. It is believed tha [illegible]idization by using both aluminum and titanium can make the small amount of remanent titanium in steel retard the growth of austenite grain in the process of heating at high temperature, and at the same time the mode and distribution of MnS inclusion can thereby be improved. Thus the sensibility to over-heating of this kind of steel is reduced. Finally, the mechanism of this steel which helps to form rock-like fracture is brought into discussion.

When steel 18Cr2Ni4WA melted by electric process (and deoxidized by using both aluminum and titanium) is changed to be melted by a mixture of open hearth and electric processes (hereafter abbreviated as open-electric melting) and (deoxidized by using aluminum alone) through the new technique of RH vacuum circulating deoxidization, in production, the production efficiency is greatly increased and the quality of steel is improved markedly. But it shows greater sensibility to over-heating and its over-heating temperature becomes as low as 100°C or so. In fracture check of this over-heating steel, the characteristics of the macroscopic fracture are as what showed in Figure 1 (plate 41). On the matrix of fiber-like fracture, there are grain-like grey cross-sections with different orientations and without metal shining and they are usually called rock-like cross-section or rock-like fracture.(1) For the purpose of finding out the reason of forming the rock-like fracture of this steel and the effective way of reducing the sensibility to over-heating for open-electric melted steel, we studied the over-heating fracture of five 18Cr2Ni4WA steels produced by different methods and observed the microstructure of fracture and the components and structure of the fine inclusions.

1. Experiment Methods

The chemical components, melting processes and deoxidization methods of steel 18Cr2Ni4WA used as testing material are shown in the following Table. Steel A and B are produced by using open-electric melting but they are deoxidized by different ways. Steel C and D are both produced by same basic electric process and deoxidized by different methods. The ingots of these four steels are weighted 3 tons. After pouring when it is still red, it is sent to primary rolling shop to be cut into square blocks of 160mm.

No.	Chemical Components %											Melting Process	Deoxidization Methods
	C	Mn	Si	P	S	Cr	Ni	W	[e]酸溶Al	Ti	N		
A	0.15	0.40	0.29	0.013	0.015	1.46	4.30	1.05	0.009		0.0080	a 平-电混炼	Use Al alone
B	0.18	0.45	0.28	0.011	0.008	1.52	4.21	1.11	0.031	0.018	未分析	平-电混炼	Use both Al and Ti
C	0.14	0.40	0.27	0.007	0.014	1.55	4.25	0.96	0.048	0.020	0.0134	b 电炉冶炼	Use both Al and Ti
D	0.16	0.41	0.27	0.015	0.007	1.44	4.38	0.95	0.060		0.0154	电炉冶炼	Use Al alone
E	0.17	0.42	0.18	0.009	0.007	1.42	4.23	1.02	0.009		未分析 d	c 电炉-电渣重熔	Electric slag parent material use Al only

Note: S and P contained in steel E are analytic value of electric slag steel parent material.

a. open-electric melting, b. electric melting, c. electric melting plus electric slag remelting, d. not analysized, e. acid dissolved

and the heating temperature of the ingots is 1280°C. The 160mm square blocks are further cut into blocks of $80mm^2$ or into sample pieces of which the diametre is 18mm used to test over-heating and fracture. And testing pieces through different austenizing temperature treatment are made into Mye (a transliteration of Chinese sound) type shock testing samples. Steel E is first melted by basic electric process and deoxidized by using aluminum only and then, after electric slag remelting, rolled into blocks of $180mm^2$, and finally it is rolled into cylinderical steel of which the diametre is 110mm used as samples.

After extraction and compound molding, the fracture is examined by using China made transmission electron microscope, and diffraction analysis of selected region is also made. Before this, the microstructure and grain situation of the testing samples are all investigated by using metallographic microscope.

2. Results of Experiment

2.1 The Sensibility of the steel to Over-heating

Steels produced by different processes are wrought into fracture testing samples of $\phi 16 \times 120$mm. After in salt bath they are respectively heating and retaining temperature at 1100, 1150, 1200, 1250, 1300 and 1350°C for 70 minutes and air cooling (first treatment), they are broken for examining their fractures. One half of them are wrought into Mve's shock testing samples and examine their tenacity value. The other half are again through temperature retaining at 950°C for 60 minutes and air cooling; again 850°C for 60 minutes and air cooling; and again 170°C for 180 minutes and air cooling, then measure their tenacity value and examine their fractures, and the results are what as shown in Figure 2.

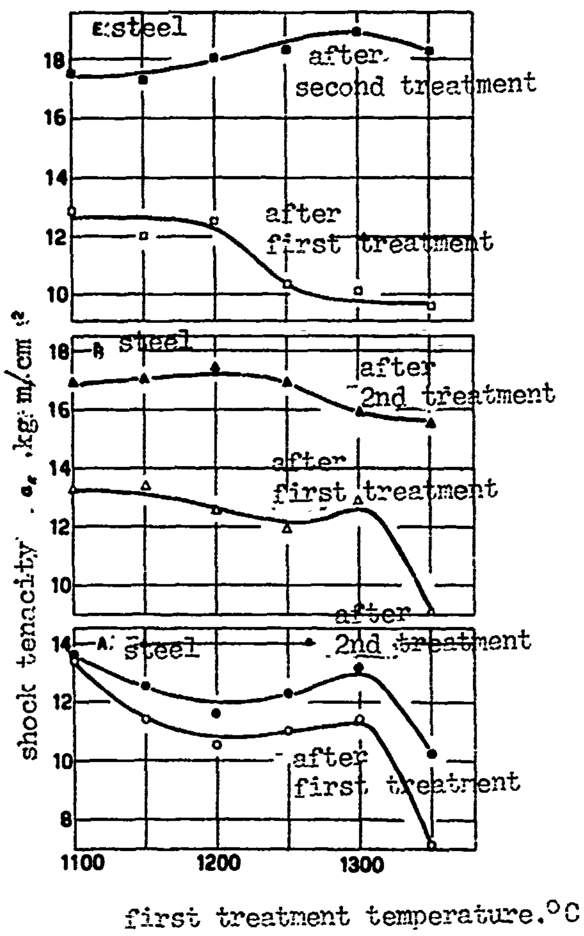

Figure 2 Comparison of sensibility to over-heating of steel A,B, and E

Of steel A, after a treatment at 1350°C, the shock tenacity value rapidly becomes low,and correspondingly the fracture becomes typical rock-like fracture and it cannot even improve after second treatment. The austenite grain begins coarsening when it is at 1200°C. The average diametre of the grain is about 2.7mm when the temperature reaches 1350°C and the greatest diametre is 4 mm or so.

Of steel B, after treatment at 1350°C, the shock tenacity value also becomes remarkably low, but it can restore after second treatment. The grain will not begin coarsening until the temperature reaches 1300°C and the fracture of the testing samples shows no over-heating small plane at 1350°C treatment.

The changes of shock tenacity value and the fracture of steel C are similar to steel B. Steel D is similar to steel A.

Of steel E, the temperature at which the shock tenacity value becomes unusually low after first treatment is 1250°C. It seems that this steel is easy to be over-heated and it will improve noticeably after second treatment. The examination of its fracture shows that it will begin to have grain-like cross-section when temperature reaches 1300°C, and its appearance looks like rock but it is not rock-like fracture and it can restore after second treatment.

2.2 The Investigation of Rock-like Fracture

Of steel A and D, although melting processes are different,however,they can both be deoxidized by using aluminum alone, and as a result they both have high sensibility to over-heating. After high temperature austenizing treatment at 1350°C or above, they both begin to have rock-like fracture. Steel A can be used as an example as shown in Figure 1 (plate 41). The metallographic testing sample, after being corroded in a mixed liquid of satuarted picric acid-soap powder-Singel (a transliteration of Chinese sound) ash, will show clear coarse austenite grain and the average size of the grain is 2.7mm. This is primary austenite grain. The boundary of these grains is easier to be corroded than that of real austenite grain (5-7) formed under state of quenching. They look black and coarse under a microscope which can amplify 100 times larger, such as the three-fork boundary in Figure 3 (plate 41).

The process by which rock-like fracture is formed can be observed from Figure 4, (plate 41). The picture was taken from the cross-section which is perpendicular to the fracture of the testing sample. The left-hand end of the picture is close to the surface of the fracture, and observation can be made by selecting some crack which is connected with a certain small over-heating plane on the surface of the fracture on testing sample. From Figure 4, it can be seen that the crack on left-hand side spreads along primary austenite grain boundary (flat-straight large grain boundary), and the crack direction evidently has no relation with the fine real austenite grain boundary formed under stste of quenching. This can prove that the over-heating small plane of rock-like fracture is the surface of primary austenite grain boundary.

The typical form of microcrack of over-heating small plane (or intercrystal small plane) of rock-like fracture is illustrated in Figure 5 and 6 (plate 42). These intercrystal small planes are formed by great amount of isometrical plastic indentations, and some very fine sphere-like inclusions scatter at the bottom of the plastic indentations. The average diametre of the plastic indentation is 1.5 micron measured from ten viewing fields selected randomly, and the average diameter of of the sphere-like inclusion is 0.3 micron. From these investigations, we can infer that the plastic indentations are formed by the very fine inclusions which precipitate on the surface of primary austenite grain boundary. In addition, on the over-heating small plane, a few larger inclusions can be found as well and they, due to deformation, are of the shape of long strips. In some local region of over-heating small plane there are anatomical and piercing cracks (micro-porous congregation type--plastic indentation) and other forms of cracks. In these microscopic regions usually there is no inclusion. The crack in the fiber-like matrix part of rock-like fracture is piercing plastic crack and there is no inclusion either.

In order to decide the type of the fine sphere-like inclusion mentioned above, we made selective region electron diffraction analysis of extraction and compound molding of rock-like fractures. The result shows that these those inclusions are MnS of cubic structure and small amount of MnS of hexagon structure. Figure 7 (plate 42) is selective region electron diffraction chart and index of cubic MnS. The dots pattern in the Figure is single crystal diffraction chart of MnS and the two diffraction rings which take the central dot as center are reflection $\{220\}$ and $\{422\}$ of MnS. The six diffraction dots with large distance among crystal planes all fall on diffraction ring {220}. The presence of these two diffraction rings shows that in the selective region, which has been in use, there is a certain amount of fine MnS inclusion.

The MnS contained in steel, in chemistry, is an instable inclusion and it can be easily corroded in 5-10% soft acid. Such a characteristic can be used for making further investigation of inclusions. Soaking carbon compound mold membrance which contains more inclusion substances into 10% nitrated alcohol solution for 24 hours, then examine it with electron microscope, the findings will be what as shown in Figure 6. The inclusions are all corroded up, so it proves that the inclusion on over-heating small plane is MnS.

2.3 The Investigation of Fractures of Steel Which Uses Both Aluminum and Titanium for Deoxidization

Steel B and C although produced by using different processes can both be deoxidized by using aluminum and titanium. When austenizing temperature reaches 1400°C, there is no sign of having serious over-heating fracture in quenching state that follows, but there are fibric fractures. Taking steel B for example, the characteristics of macroscopic fractures are as shown in Figure 8 (plate 43). Metallograhpic observation indicates that the average size of primary austenite grain is only 0.35mm. The electron microscope observation of extracted compound mold indicates that these fibric fractures are formed by large and long plastic indentations. At the bottom of isometrical plstic indentation, there are fine MnS inclusions (about 0.2-0.3

micron), of which the shape is different as shown in Figure 9 (plate 43). Through selective region electron diffraction examination, they are identified as AlN. But in steel A, there has never been any fine sphere-like MnS deposit on over-heating small plane.

In addition, in the relatively even region on the macroscope of fibric fracture, there is another kind of fine deposits and most of them are block shaped. Through selective region electron diffraction examination, they are identified as TiN. But the amount of this kind of TiN particles on fibric fracture of steel B and C is small. In order to find out the rules of TiN distribution in steel, we examined the fracture of temper brittleness. After having second treatment, steel B is tempered again for three hours, then break it, we find fracture of temper brittleness and extracted carbon compound mold membrance. When they are examined again by using electron microscope, there are more fine TiN particles. These TiN deposits often scatter on the surface of primary austenite grain boundary, and that is the position of cracks along the grain on the fracture, as illustrated in Figure 10 and 11 (plate 43, 44). But at the position of anatomical piercing fracture (as the right-lower part of Figure 10) and the position of plastic piercing fracture (as the right-lower part of Figure 11), no TiN deposit comes out. A selective region diffraction analysis is made for TiN particle, and Figure 12 (plate 44) gives its electron diffraction chart and index. From Figure 12, we can see diffraction rings which are not clear and sharp, and from TiN deposit on fracture and its electron diffraction chart of steel B, we can as well see diffraction rings. In some picture, the diffraction rings are clearer. They can be identified as corresponding grain surface reflection of TiN. At the same time, in the electron diffraction chart, diffraction dots and diffraction rings are marked in selective region. Besides the TiN particles of 0.2-0.4 micron, there are many still smaller TiN particles which are about a few hundred angstrom. In short, in steel which can be deoxidized by using both aluminum and titanium, there is great amount of TiN particles and they often scatter at primary austenite grain boundary. According to their size, they can be classified into two different kinds: one size is 0.2-0.4 micron and they are of block shape, and the other size is only a few hundred angstrom and they scatter among block-shaped TiN.

2.4 The Characteristics of Fractures of Electric-electric Slag Steel

After first treatment at 1350°C, the macrofracture of steel E generally appears to be granular cross-section. The electron microscope examination after its extraction compound molding indicates that the cracking process of this steel takes place either along crystal plasticity, as shown in Figure 13 (plate 45) or along crystal brittleness, as shown in Figure 14 (plate 45). So we can know that this kind of granular cross-section is the surface of grain boundary formed at the time of high temperature austenization. On these microsections, there is no MnS inclusion. So it is sharply different from the rock-like fracture of steel A.

After second treatment, the characteristics of granular cross-section of steel E is remarkably improved. When it is broken, there are typical curve fibric fractures and their characteristic is a typical piercing plastic crack and it is made of extended plastic indentations.

High grade metallographic examination indicates that oxide inclusion and sulphide inclusion in steel E are all at 0.5 level. The oxide inclusion of steel A and D is at 1 level and sulphide inclusion is at 1.5 level. Therefore, they can all be deoxidized by using aluminum alone, but of steel E, after electric slag remelting, the inclusion is reduced graetly and the grain boundary becomes pure and clear.

3. Discussions

3.1 The Mechanism of Rock-like Fracture

In some research report,(1-4) there are discussions on problems of overheat and over-burn of alloy structural steel, and on the formation of rock-like fracture suggest different opinions, too. In the following, we shall try to make an analysis of our observations of steel 18Cr2Ni4Wa rock-like fracture.

Of MnS contained in steel, the solubility in austenite will increase as the heating temperature of the steel rises.(1) The higher the temperature is, the less the remanent nascent MnS, which was not dissolved, will be. Experiment shows that in the process of cooling after austenizing, MnS, which has been dissolved in steel, will become shpere-like particles and again deposit on the surface of primary austenite grain boundary. The higher the austenizing temperature is, the more the MnS particles deposited on the surface of grain boundary will be. On the other hand, when steel is austenized at high temperature, the higher the temperature of heating is, the greater the tendency of austenite grain growth will be. When it is heating at a temperature above over-heating, the grain will become specially coarse, and the surface of grain boundary will turn to be flat surface (see Figure 3 and 4, plate 41). Once a great amount of fine MnS is deposited, the connecting strength of grain boundary becomes seriously weak and thereby the ability of fracture resistance of primary austenite grain boundary becomes low, so it becomes easy for fractures to spread out. So after the tenacity of matrix of the steel is strengthened due to quenching (or modification), fractures will easy occur along the surface of primary austenite grain boundary. Thus on the matrix of fibric fracture comes out many over-heating small planes, and they are small planes at primary austenite grain boundary. Then they develop into rock-like fractures. Because the general heat treatment cannot make the primary austenite grain fine again and also because the dissolution of MnS can take place only at temperature above 1200°C, it cannot use heat treatment of low temperature to redissolve it and change its distribution pattern. The second treatment therefore cannot improve the rock-like fracture. But rock-like fracture is an unpermissible defect of alloy structural steel.

A comparison of steel E, A and D indicates that when the steel is heating at high temperature, the austenite grains of three different kinds of steel will similarly become coarse. But through electric slag remelting, steel E becomes relatively pure and there is no great amount of deposit of MnS inclusion on the surface of grain boundary so generally there is no rock-like fracture. The shock tenacity of steel E after first treatment shows greater senibility to over-heating, but after second treatment it improves to great extent.

So we think that there must be two conditions for alloy structural steel to form rock-like fracture: first, when the steel is austenizing at high temperature, it attains primary austenite grains which are coarse enough, and secondly, the steel contains a certain amount of MnS inclusion, much of which are dissolved when the steel is in the process of heating at high temperature, and later in the process of cooling, they become shpere-shaped MnS and again deposit on the surface of primary austenite grain boundary. The presence of such flat and straight surface of primary austenite grain boundary which contains great amnout of fine MnS inclusion is the main reason of forming rock-like fracture.

3.2 The Effect of Small Amount of Aluminum and Titanium on the Sensibility of Steel to Over-heating

It has been pointed out in some documents (5-7) that when the amount of acid-dissolved aluminum is 0.02-0.05%, after heat working and heat treatment, there will be fine AlN deposit and it will have favorable effect on delaying the process of recrystalization and fining austenite grain. But our experiments as we report in this article show that in steel D the amount of acid-dissolved aluminum (0.060%) is much higher than in steel A (0.009%). But when austenizing is made at high temperature, the situations of grain-coarsening of these two steels are almost the same. After heating at a temperature above 1300°C, these two steels will both produce over-heating. This proves that the increase of acid-dissolved aluminum in steel will not have favorable effect on over-heating of alloy structural steel. This is because when steel is heated at a temperature above 1200°C, AIN will be dissolved solidly and the favorable action of AIN in the steel as mentioned above will not show. Therefore, steel A and D, which can be deoxidized by using aluminum alone, have equally high sensibility to over-heating although their amounts of acid-dissolved aluminum are different.

When the temperature of austenizing of steel B and C reaches 1400°C, the average size of austenite grain is still 0.35mm. This can be explained by the effect of small amount of remanent titanium. The content of remanent titanium in these two steels is 0.01-0.02% and they also contain about 0.01% of helium. The examination of extracted compound molding by using electron microscope indicates that at primary austenite grain boundary, there is a great amount of fine TiN deposit. These TiN come out after the steel ingot has solidified in the process of cooling. They have higher stability than AIN, and they can exist without being dissolved in a range of high temperature.(8) And because TiN particles scatter among grains, in the process of high temperature austenizing treatment of the steel, they can very effectively retard the growth of austenite grain. This is the reason that a small amount of titanium can make the grains fine.

Our experiments show that steel (B and C) which contains small amount of titanium, after high temperature austenizing treatment even at 1400°C, will have no fine MnS deposit on the extracted compound mold of fracture. But, occasionally some MnS inclusion which is long, not dissolved and in large pieces can be seen. The contents of S and Mn in steels (A and D) which do not contain even small amount of titanium are close to those of steel B and C. But on the fracture, after first treatment at 1300°C, there are many fine MnS particles deposited at primary austenite grain boundary. Thus we think that a small amnout of remanent titanium has changed the rules of dissolution and deposit of MnS in the process of high temperature austenizing, lowered the solubility of S in steel, improved the form and distribution situation of sulphide in steel and consequently produced some favorable effect on lowering the sensibility of steel to over-heating. On the other hand, because TiN inclusion can effectively retard the growth of high temperature austenite grain, the total area of grain boundary of fine grain steel is much larger than that of coarse grain steel. Even the total number of MnS particles deposited at grain boundary is close, the number of MnS particle (density) on a unit arae at grain boundary of fine grain steel is noticeably low. This is also very good to prevent the occurrence of rock-like fracture.

In summary, using proper amount of aluminum and titanium for deoxidization results in that there is a small amount of titanium remains in the steel. Obviously it has preventive function to the forming of over-heating as well as the occurrence of rock-like fracture or helps to delay the happening until there is a much higher temperature of austenizing heating. Thus the sensibility of the steel to over-heating is effectively lowered.

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