BEHAVIOR OF TI(C,N) PARTICLES IN C22+AL+TI STEEL DURING HEAT TREATMENT

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Introduction

Acicular ferrite has been successfully applied for improvement of toughness of weld metals in welding industry for many years due to an attractive combination of high strength and good toughness with respect to its refined grain size and interwoven structure [1-5]. The first stage of its investigation [6, 7] is tied up with investigation of weld joints microstructure [2, 8]. Structure of weld joint can consist of acicular ferrite regions due to one of the main conditions of acicular ferrite formation is presented in these regions: content of nonmetallic inclusions with size more than 50 nm is higher than 10^{18} m^{-3}, and average size of inclusion is~ 0,4 µm [8]. These inclusions are rather uniformly distributed in weld seam and they could act as effective nucleation sites for heterogeneous nucleation of acicular ferrite plates under condition that this region of weld seam is cooled with rates sufficient for bainitic transformation. In structure of weld seam such inclusions are mainly presented with refractory oxides [9]. It has been recognized that acicular ferrite is hardly observed in wrought steel because wrought steels have a much lower density of non-metallic inclusions than weld metals. [5].

It has received much attention that there are particular non-metallic inclusions effective for producing acicular ferrite in HAZs which inherit the composition of wrought steels. In recent years, several attempts have been made to produce acicular ferrite microstructure in wrought steels by utilizing non-metallic inclusions as nucleation sites for acicular ferrite plates [9-11].

Morphology and dimensions of non-metallic particles play important role in acicular ferrite structure formation along with their distribution density [12]. It should be mentioned that complex particles of refractory metals (Ti_XO_Y, TiN, Ti(C,N), Nb(C,N) etc.) with sulfides (especially MnS) could be highly preferred from acicular ferrite plates inoculation point of view [13]. Therefore they provide not only good ferrite/inoculation lattice correspondence but also promote intragranular ferrite plates nucleation, by means of austenite local volumes depleting manganese [12].

Data about the mechanism of acicular ferrite structure formation are rather contradictory [5,8,10,12,14-16]. There are two paradigms regarding the formation of acicular ferrite. One is based on the first stage being rapid diffusionless growth of acicular ferrite and the subsequent formation of carbide occurring by precipitation of the supersaturated ferrite. The other paradigm is based on the first stage being the formation of acicular ferrite under carbon diffusion and on the subsequent simultaneous growth of carbide and ferrite side by side.

Every type of phase transformation has a number of specific features. In particular during shear transformation structure of steel is characterized with strong

oversaturation of ferrous solid solution with carbon. During subsequent heating carbon may diffuse from the – ferrite lattice and form carbides [17,18]. Thus martensite or bainite dissociation followed by carbides precipitation is the main process during tempering of quenched steels.

Lately a series of experiments has been held to establish ability of Al-Ti-N alloying system on formation of acicular ferrite structure in C-Mn wrought steel. It has been found that Al-Ti-N alloying along with heat treatment promotes acicular ferrite formation in basic structural C-Mn steel (C22 by DIN standard) [19-21]. This happens because obtained inclusions of Ti(C,N) and MnS along with AlN act as effective point nucleation sites for acicular ferrite plates inside the prior austenite grain boundaries. Structure and properties of the investigated steel have been studied [19-22]. In this sense investigation of the carbonitride phase of structural steel 22 microalloyed with Al+Ti+N could allow us to identify the composition and distribution of carbonitrides in steel.

Information about tempering of low carbon steels with acicular ferrite structure is completely absent. Thereby microstructure investigation of steel with the structure of acicular ferrite after various tempering modes has been held in order to establish the allocation of interstitial carbon in – ferrite (lattice) and study its movement during tempering, and also determine the relevance of tempering operation of acicular ferrite in structural steel inoculated with nitride formative elements.

Experimental Technique

Two 20 ton ingots of low carbon-manganese steel inoculated by Al-Ti-N have been melted in electric-arc furnace. In EN and DIN standards the closest by chemical composition designation for the investigated steel is C22 additionally inoculated by Al and Ti (DIN 1.0427). The chemical composition of the investigated steel is presented in Table 1.

Table 1.

Chemical Composition of the Investigated Steel.

Steel 22+Al+Ti	Wt. %, Fe-balance									
	C	M	Si	P	S	C	Cu	Al	Ti	N
	0.19	0.63	0.28	0.015	0.014	0.18	0.20	0.029	0.003	0.019

It should be mentioned that such expensive alloying elements as vanadium, niobium and molybdenum have not been alloyed in the investigated steel.

Ingots have been rolled into 60 mm square semi-finished product. Heat treatment aimed for acicular ferrite structure formation has been made.

Specimens of steel with acicular ferrite structure have been subjected to tempering. The first procedure of tempering included heating at the temperature 650^0C during 2 hours. And the second procedure involved heating at the temperature 650^0C during 4 hours.

Investigation of acicular ferrite structure after tempering has been made using scanning electron microscope. Determination of chemical composition of the investigated carbides has been held using EDX+WDX spectrometer. Character of

carbide distribution in structure of acicular ferrite has been studied with the help of the InLens SE detector of secondary electrons of the scanning microscope.

Heating of steel with titan carbonitrides and their observation has been held using confocal laser scanning high-temperature microscope which allows to make in-situ observation of metals and slags at heating up to 1700^0C with high heating rates up to 50 K/s , heating and cooling in vacuum or gas protective atmosphere, accuracy 0.25 μm, laser: He-Ne laser (=633 nm, =1.5 W); heating has been held from ambient temperature up to 1470^0C.

Investigation of titan carbonitrides morphology before and after heating has been held using light microscope Neophot 2 and also in scanning electron microscope Karl Zeiss. Determination of chemical composition of investigated particles has been held using EDX+WDX spectrometer for SEM of EDAX Company.

Quantitative analysis of titan carbonitrides particles size change after heating has been held on Axio Vert software of Carl Zeiss Company.

Results and Discussion
I. Structure of Acicular Ferrite and Ti(C,N) Particles During Tempering Process.

Specimens of C22+Al+Ti steel with acicular ferrite structure have been subjected to tempering at 650^0C temperature. Microstructures of acicular ferrite structure and acicular ferrite after tempering in 22 +Al+Ti steel are presented in fig. 1 and 2.
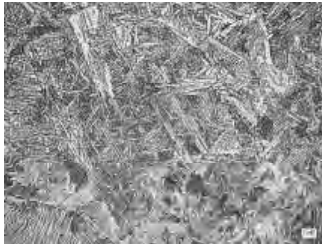

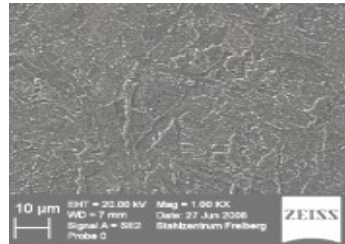Fig. 1. Acicular ferrite struct 1000 (LM).   Fig. 2. Acicular ferrite structure after 650^0C tempering, 1000 (SEM).

As it comes out from the comparison of microstructures in fig. 1 and 2, tempering leads to significant change in microstructure. Precipitation of bright particles on the boundaries of acicular ferrite plates could be observed in structure of tempered acicular ferrite. These bright particles clearly seen in fig. 3 could probably be carbides, precipitated during tempering. Effect of precipitation of carbides is characteristic for tempering of martensite when diffusion of carbon from supersaturated with it martensite plates takes place.
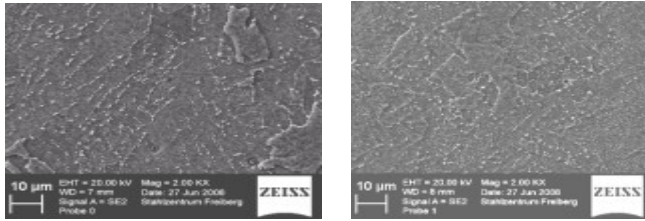
Fig. 3. Microstructure of acicular ferrite after high tempering during) 4 hours and b) 2 hours, 2000.

EDX analysis has been applied for the identification of precipitated particles. The characteristic particle along with its EDX spectrum is presented on the fig. 4. We may state that this particle is the cementite Fe_3C according to results of EDX analysis.
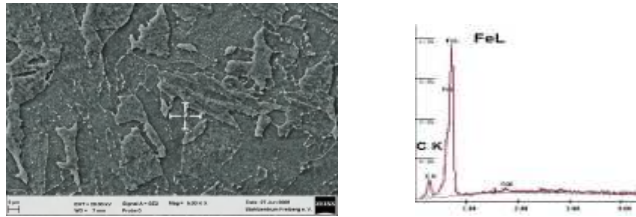

Fig. 4. Microstructure of acicular ferrite after 4 hours soaking and iron carbide particle, 5000.

InLens analysis of secondary electrons has been made in order to define preferable location of precipitated carbides. Distribution of carbides in structure of acicular ferrite after 2 and 4 hours of tempering is presented on fig. 5.
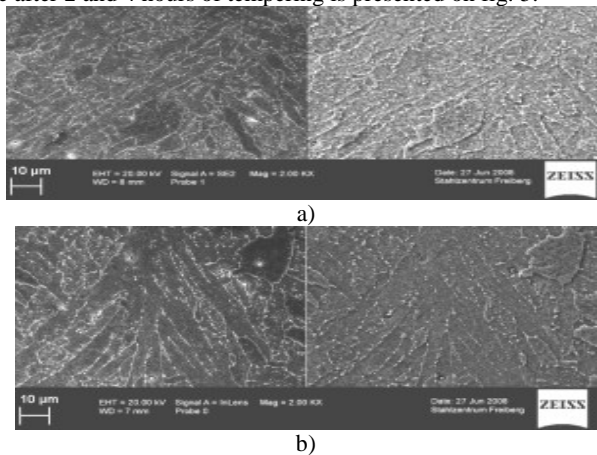

a)

b)

Fig. 5. Structure of acicular ferrite after soaking) 2 hours and b) 4 hours, InLens SE analysis, 2000.

As it comes out of structures analysis on the fig. 5, acicular ferrite laths are decorated with precipitated carbides. It could be connected with high temperature of tempering and its relatively long duration.

Increase of tempering length from 2 to 4 hours does not lead to significant structural change.

Thus the fact of carbides precipitation on the boundaries of acicular ferrite laths proved that the laths of acicular ferrite contain excessive quantity of carbon at forming of acicular ferrite structure. This redundant carbon precipitates into ferritic carbides during the subsequent tempering. (similar to martensite tempering in low-carbon steels)

On the basis of obtained data we may suppose that during formation of acicular ferrite structure the carbon is partially left inside acicular ferrite laths and partially pressed back with the front of the growing lath into the interlath space, thus saturating austenite with carbon. Apparently, given volumes of austenite will transform into martensite. Later the carbon can segregate both from acicular ferrite plates and from the high carbon martensite regions. Thus the paradigm about shear transformation character of acicular ferrite structure formation finds experimental evidence. Any significant change in size and morphology of Ti(C,N) particles during tempering process has not been observed. This is predictable due to high thermal stability of Ti(C,N) particles within $20 - 650\ ^0C$ interval (See. Fig.7a and Fig.7b)

II. Behavior of Titan Carbonitrides at Heating in Confocal Laser Scanning High Temperature Microscope.

Titan carbonitrides in C22+Ti+N steel have been investigated. Our attention has been devoted to behavior of Ti(C,N) particles during heating because in our previous works they were supposed to be effective point nucleation sites for acicular ferrite plates and effective hardening elements in structure of the investigated steel. The investigated steel has been subjected to a series of heat treatments involving heating to the temperature higher than Ac3, quenching and subsequent heating for tempering to the temperature 650^0C. Heating operations may lead to dissociation of Ti(C,N) particles that is why we investigated their behavior during heating up to the temperatures close to the melting point.

Results of investigations of titan carbonitrides behavior during heating up to 1470^0 C are presented on fig. 6 – 12.
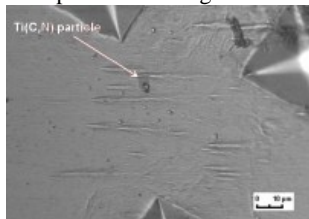

Fig. 6. Ti(C,N) particle in light microscope before heating, x1000

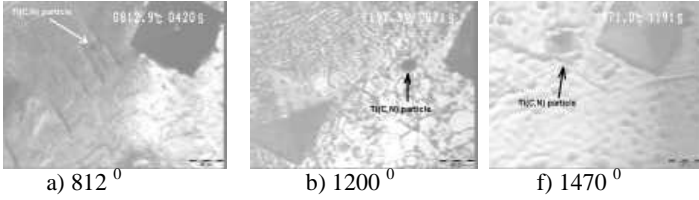Fig. 7. The same Ti(C,N) particle after heating and cooling

a) 812 ^{0} b) 1200 ^{0} f) 1470 ^{0}

Fig. 8. One Ti(C,N) particle in laser microscope during continuous heating at
a) 812^0 , b) 1200 ^{0} and c) 1470 ^{0} ; x500.

Fig.9 Another Ti(C,N) particle
before heating

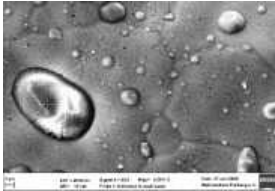

Fig. 10 Spectrum of the given particle

Fig. 11 Ti(C,N) particle after
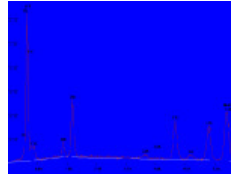heating and cooling

Fig. 12 Spectrum of the given particle

As it follows from the figures coarse particles of titan carbonitrides do not essentially change in size after heating from the room temperature up to 1470 ^{0}C and subsequent cooling. Investigation of the particle after over crystallization allows us to make a suggestion that non-dissociated particle of Ti(C,N) in liquid steel acts as a substrate for Si, Ca Cr, Mn bonds segregation.

In order to investigate the process of Ti(C,N) dissociation, the area of the particles before and after heating has been measured. Typical particles are presented on fig. 13 – 16.
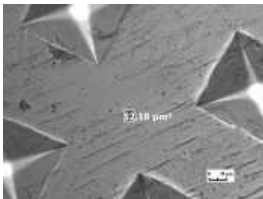
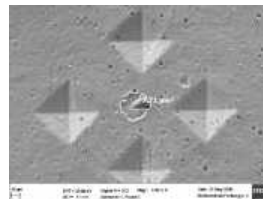

Fig. 13 I particle before
heating, x500

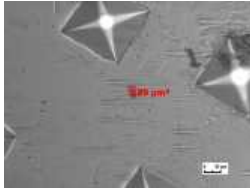Fig. 14 I particle after heating
to 1450^{0}C, x1000

Fig. 15 II particle before heating, x500 Fig. 16 II particle after heating to 1470^{0}C, x1000

Obtained data shows that at heating the square section area of the particle reduces on 36 % in case of the first (I) typical particle (from 12,18 micrometers^2 to 7,71 micrometers^2) and on 33% in case of the second (II) typical particle (from 7,89 micrometers^2 to 5,1 micrometers^2)

Conclusions

1. It has been experimentally proved that melting of steel with Ti(C,N) particles does not lead to complete dissociation of Ti(C,N).

2. At the same time partial dissociation of Ti(C,N) particles occurs. The size of the particles decreases on 33-36 % at 1450 - 1470^{0}C.

3. Given particles act as the substrate for different compounds of liquid steel association during recrystallization.

4. It has been established that any significant changes in size and morphology of Ti(C,N) particles during tempering process do not occur. It has been obtained that tempering of acicular ferrite leads to precipitation of carbides. It has been experimentally proved that precipitated carbides are ferric carbides with expected formula Fe$_3$C. Analysis of preferable places of location of carbide particles precipitation has been held. It has been shown that precipitated carbides are predominantly situated on boundaries of acicular ferrite laths.

5. It has been experimentally proved that laths of acicular ferrite just after quenching are supersaturated by carbon atoms.

6. It has been shown that increase of soaking duration from 2 to 4 hours does not lead to significant change in structure of tempered acicular ferrite.

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